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Evaluation of Stationary Source Particulate Measurement Methods

Volume IV. Basic Oxygen Furnaces

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16. ABSTRACT

A procedure, EPA Method 5, for sampling and determining particulate concentrations in emissions from stationary sources was specifically evaluated at basic oxygen furnaces (BOF) equipped with wet-scrubbers or electrostatic precipitator (ESP) controls. Although wet-scrubber emissions could potentially present the most difficulty since the sampling system must handle a moisture-laden gas stream with entrained water droplets, no problems were found when using Method 5. Variation of the sampling rate at 0.7 and 1.3 times isokinetic also did not significantly affect the accuracy of the mass measurements. Use of a higher purity, lower pH filter medium (ADL quartz) gave mass results that were not statistically different than those obtained with MSA 1106 BH, the commonly used filter material.

Chemical analyses confirmed that the Method 5 procedure extracts a representative sample of the BOF/ESP stack particulate emissions. The same general chemical composition was found on Method 5 filters and in grab samples removed from the stack at the sampling point.

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Volume IV. Basic Oxygen Furnaces

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ABSTRACT

The work described in this report is part of a study to evaluate the EPA Method 5 procedure for sampling a variety of stationary sources. Specifically, this study addresses the reliability of this reference method for measurement of particulate emissions from basic oxygen furnaces (BOF). The facilities at which sampling was performed are equipped with emission controls which are representative of the two principal systems used to abate BOF emissions, namely, high-energy wet scrubbers and electrostatic precipitators.

Experiments were conducted to study the effects of sampling system temperature, filter material, and anisokinetic sampling on mass results obtained with Method 5. In-stack sampling was used as a comparative technique to assess the Method 5 procedure. Chemical and limited physical characterizations were performed to evaluate the representativeness of the collected particulates and to identify variations which lead or may lead to mass differences introduced by various sampling parameters. Gaseous emissions were analyzed to identify species which may interact with the particulate sampling process.

Operationally, the Method 5 procedure performed satisfactorily when used to sample particulate emissions from the wet scrubber-equipped BOF. Although some fractionation of certain species in the sampling train was observed, the general chemical composition of the Method 5 collections were representative of the stack emissions. Experiments in which sampling was performed at anisokinetic rates (0.7 and 1.3 times isokinetic), and sampling system temperature was varied from 84 to 191°C, did not show observable differences in mass loading results.

Problems were encountered in the use of an in-stack method for sampling the wet scrubber emissions. The moisture-ladened stack gas with entrained droplets saturated the filter with water causing a high pressure drop and rupture of the filter. Isolation and heating of the filterholder were required to keep the filter dry. Although the results of the in-stack Method 5 comparison were scattered, the data indicate that in-stack sampling may give higher mass loading measurements.

Experiments at the BOF equipped with electrostatic precipitators indicate that Method 5 gives reliable results and provides collections which are representative of the stack emissions. Differences in mass loading measurements were not observed when sampling was performed with different filter materials (glass fiber and quartz) or when the sampling system temperature was increased to stack gas temperature (∿177°C). A limited number of experiments indicates that in-stack sampling may yield lower mass results than Method 5.

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SECTION 1

INTRODUCTION

The Clean Air Act as amended in 1970 provides the impetus for programs to improve the air quality in the U.S. through research to broaden the understanding of the effects of air pollutants, research and development of techniques to control emissions, and the enactment of air quality regulations to protect the public welfare. Pursuant to Section 111 of the Act, the Environmental Protection Agency (EPA) on December 23, 1971, promulgated Standards of Performance for New Stationary Sources (amended) for fossil fuel-fired steam generators, incinerators, Portland cement plants, and nitric and sulfuric acid plants.(1) On March 8, 1974, similar performance standards were issued for asphalt concrete plants, petroleum refineries, storage vessels for petroleum liquids, secondary lead smelters, secondary brass and bronze ingot production plants, iron and steel plants, and sewage treatment plants.(2) All new and modified sources in the preceding categories are required to demonstrate compliance with the standards of performance.

The performance standards are intended to reflect "the degree of emission limitation achievable through the application of the best system of emission reduction which (taking into account the cost of achieving such reduction) the Administrator determines has been adequately demonstrated".(3)

Compliance with required performance is determined by testing procedures specified with the standards. The use of a procedure called "Method 5 Determination of Particulate Emissions from Stationary Sources"(4) is specified in all instances where particulate mass emission measurements must be made. The Method 5 procedure consists of isokinetic extraction of a sample from the emission stream with a heated probe and collection of the particulates on a heated filter. With the recent exception of fossil fuel-fired power plants(5), the same sampling system operating parameters have been adopted for all stationary sources.

The source categories subject to Method 5 particulate measurements include diverse processes which encompass a wide range of the following emission characteristics; moisture content, gas temperature, gas composition, particulate concentration and composition, and flow dynamics. Interaction of these emission properties with the Method 5 sampling technique can produce significant variations in the results of particulate emission measurements. The following are examples of some of the reactions which may affect particulate measurements:

(1) SO_3 or H_2SO_4 in emissions can condense to form sulfates which increase the mass of collected "particulates". The SO_3-H_2SO_4 dew point is dependent on SO_3 concentration and moisture content of the emissions.

(2) The filter particulate catch may present a surface for reactions with gaseous emission components such as SO_x and NO_x. Reactivity would be dependent on particulate loading and composition and on gas composition of the emissions.

(3) Changes in gas temperature in the sampling system may alter the apparent particulate concentration through condensation or evaporation.

Such interactions with the sampling process must be recognized and controlled if Method 5 is expected to yield reliable particulate measurements for individual source categories.

The work presented in this report was performed as part of an EPA program to study the applicability of the Method 5 procedure to measurement of particulate emissions from a variety of stationary sources. Specifically, this work addresses the question of whether Method 5 provides an accurate, reliable measurement of particulate emissions from basic oxygen furnaces (BOF). The study included BOF facilities with the two types of emission controls in common use, wet scrubbers and electrostatic precipitators.

Volume I in this series covers a similar study of Portland Cement Plants[6] and Volume II Oil Fired Power Plants[7].

SECTION 2

CONCLUSIONS

The results of this study lead to the following conclusions regarding methodology for measuring mass emissions from basic oxygen steelmaking furnaces (BOF).

BOF WITH WET SCRUBBER

- Method 5 as promulgated in the Federal Register, December 23, 1971, appears to be a reliable procedure for particulate mass emission measurements. The precision (repeatability) of mass measurements made by concurrent sampling with two systems is estimated to be about 2.8 percent. Chemical analyses show that although fractionation of some species may occur between the probe and filter collections, the composition of the total system catch is representative of the stack emissions.

- Mass emission results obtained with Method 5 are not significantly affected by sampling variations at 0.7 and 1.3 times isokinetic rate. Particle size measurements of the emissions indicate a mass mean diameter of about 0.2 μm. Consequently, anisokinetic sampling would be expected to have little effect on the mass measurements.

- Variation in sampling system operating temperature was found to have no effect on mass emission measurements. Experiments in which sampling was performed with Method 5 probe outlet gas and box filter temperatures of about 84, 149, and 191°C gave essentially the same results as obtained with a Method 5 train operated at the minimum specified temperature, 121°C.

- Operational problems were encountered in use of in-stack sampling on the wet scrubber. Isolation and external heating of the filterholder are required to prevent saturation of the filter with water. Even with these measures, it was difficult to regulate the in-stack filter temperature to prevent collection of moisture.

- Comparisons of in-stack and Method 5 show a considerable amount of scatter; however, generally results indicate that in-stack sampling may give higher mass emission data.

BOF WITH ELECTROSTATIC PRECIPITATOR

- Method 5 as promulgated in the Federal Register, December 23, 1971, appears to be a reliable procedure for measurement of particulate mass emissions. The repeatability of measurements made by concurrent sampling with two trains is estimated to be about 3.4 percent. Chemical analysis

confirm that samples collected with the Method 5 procedure are representative of the in-stack particulate composition.

- Statistically designed experiments show that there is not a significant difference between mass results obtained with Method 5 when two different filter materials are used and when the sampling train is operated at stack gas temperature. There were no statistically significant difference in results obtained with MSA 1106BH and ADL quartz filter media. Results from experiments in which sampling trains were operated at stack temperature, ∿177°C, were not statistically different from results obtained with a sampling system temperature of 121°C, the minimum specified in Method 5.

- In-stack sampling appears to yield mass emission measurements which are lower than those obtained with Method 5. Experiments with use of MSA 1106BH flat filters and thimbles marketed by Carborundum Company for in-stack particulate collection gave results which were, on the average, about 25 and 16 percent lower, respectively, than Method 5 values.

- Particle size measurements indicate a mass mean diameter of 4 μm as compared to 0.2 μm for the wet scrubber. These results are consistent with these types of control devices.

SECTION 3

RECOMMENDATIONS

The study demonstrates that particulate mass emissions from basic oxygen furnaces both with wet scrubber and electrostatic precipitators can be determined with acceptable precision and representativeness with Method 5 as currently promulgated. Therefore, major revisions in Method 5 when applied to BOF facilities are neither necessary nor recommended.

The use of in-stack sampling for wet-scrubber installations is not recommended. When used without external heating, the in-stack filter becomes saturated with water leading to restricted flow rates. When external heating is used, considerable difficulty is encountered in controlling the filterholder temperature.

Operationally, there are no apparent problems in the use of in-stack sampling techniques for ESP equipped BOF facilities. However, additional studies are recommended to determine if in-stack techniques give results equivalent to Method 5.

SECTION 4

EXPERIMENTAL WORK AND RESULTS

EXPERIMENTAL APPROACH

In line with the program objectives, the experimental plan was formulated (1) to study parameters of Method 5 which might affect particulate mass measurements, (2) to characterize the emissions, both particulate and gaseous, to identify reactive species which might affect sampling results, and (3) to compare Method 5 with in-stack sampling techniques.

Sampling parameters which were studied included filter material, sampling system temperature, and deviation from isokinetic sampling rate. These studies were intended to reveal the sensitivity of particulate measurements to the sampling variables and to determine if current Method 5 operating parameters are within a range which will produce accurate, reliable results.

Various in-stack filter configurations were compared with Method 5. Particulates collected under in-stack conditions might be considered less subject to compositional alterations, especially by condensation products and reactions which may occur upon cooling below stack temperature assuming stack temperature is greater than 121°C (250°F). Accordingly, comparison of the in-stack and Method 5 particulate collections provides an approach to study of sample alterations which may be introduced by the Method 5 sampling procedure. In addition, in-stack collection essentially eliminates deposition of the particulates in the probe and facilitates the study of reactions which may occur in the probe, e.g., condensation of H_2SO_4 or organic materials.

Another consideration behind the methods comparison is that EPA is considering adoption of the in-stack technique as an optional performance test method. Equivalency of Method 5 and the in-stack method must be demonstrated to maintain consistency with established performance standards.

The approach selected to conduct the experimental study consisted of concurrent sampling at approximately the same point in the BOF emissions stack with two sampling systems operated in various sampling configurations and under the various conditions under study. Filter and probe collections were analyzed gravimetrically and chemically to detect differences resulting from various sampling parameters. Analyses of the gas composition of the emissions were performed to identify components which might interact with the sampling process. Where possible, the mass emission data from the experiments were analyzed statistically to determine the significance of observed differences.

PROCESS AND SAMPLING SITE DESCRIPTIONS

Basic Oxygen Steel Process

In 1974, the production of steel in the U.S. totaled 145,720,000 tons. Production over the past 4 years, a relatively weak period economically, has increased at a rate of about 2 percent per year. In 1974, carbon steel accounted for 86.9 percent of the total production with alloy and stainless steels accounting for 11.6 and 1.5 percent respectively.

The principal steelmaking process used in the U.S. is the basic oxygen furnace (BOF) process. Of the total 1974 production, 56 percent of the steel was made by this method. Open hearth and electric furnace processes produced 24 and 20 percent, respectively. In the past 3 to 4 years, steelmaking by the open hearth process has leveled off, while basic oxygen steelmaking has continued to increase by 29 percent.

The basic oxygen process or "BOF process" as it is commonly called is a batch reactor process wherein hot metal and scrap are charged into the furnace, and lime, fluorspar, plus other fluxes are added and these are reacted with oxygen which oxidizes out the major impurities--principally carbon, phosphorus, silicon, and magnesium--to form a low-carbon steel. Under ideal conditions, the entire operation cycle takes 35 minutes and includes the sequence of operations listed in Table 1.

TABLE 1. OPERATIONAL STEPS IN BOF PROCESS

(1)	Scrap metal charge is loaded in vessel.
(2)	Hot metal (pig iron) from the blast furnace is added to complete the charge.
(3)	The lance is lowered near the bath and the oxygen blow is initiated.
(4)	Flux is added.
(5)	Blow is terminated after about 20 minutes.
(6)	Temperature of the steel is measured and a sample is taken for chemical analysis.
(7)	Reblowing may be necessary to achieve desired chemical composition and/or temperature.
(8)	Steel is teemed into ladle.
(9)	Slag is discharged.

Particulate emissions from the BOF process includes metal, slag, and impurities swept from the vessel by the supersonic oxygen stream and the violent exothermic reaction of the oxygen with the impurities in the pig iron. Gaseous emissions are high in CO and CO_2 resulting from oxidation of the carbon in the pig iron. Some form of emission control device is required to bring particulate emissions into compliance with EPA Performance Standards. Wet scrubbers or electrostatic precipitators are the types of control systems which are commonly used.

Sampling Site--Wet Scrubber Control

Sampling experiments involving a wet scrubber emission control system were performed at a modern BOF factory. The facility has two 200-ton vessels, only one of which is in operation at any one time. The charge to the furnace consists of approximately 50 tons of scrap and 150 tons of hot metal. Scrap preheating is not used.

The emission control system utilizes a high energy wet scrubber gas cleaning process illustrated in the simplified flow diagram, Figure 1. The system includes a movable skirt which seals over the vessel during the blow to conduct particulates and hot gases to a quench section where they are sprayed with water causing the coarse particulates to drop out. Then the gases go through a secondary venturi where a large pressure drop provides additional particulate removal. The gas, then cooled to a temperature of about 65°C (150°F), goes through the fan and up the stack where a pilot burner ignites the CO present in combustible quantities. The wet scrubbing process is designed to operate at about 99.98 percent efficiency.

At the initiation of the blow and near its termination, the head space above the vessel is purged with nitrogen to sweep oxygen from the emission control system.

The emissions to the stack are heavily moisture-laden and have the following characteristics.

Composition

CO - 70 to 75 percent
CO_2 - 10 to 20 percent
O_2 - 0.1 to 0.3 percent
NO_x - $\sim$1000 ppm max during N_2 purge
SO_2 - negligible
particulates - <50 mg/Nm^3
moisture - saturated with entrained droplets

Velocity

8 to 13 m/sec

Temperature

49 to 65°C

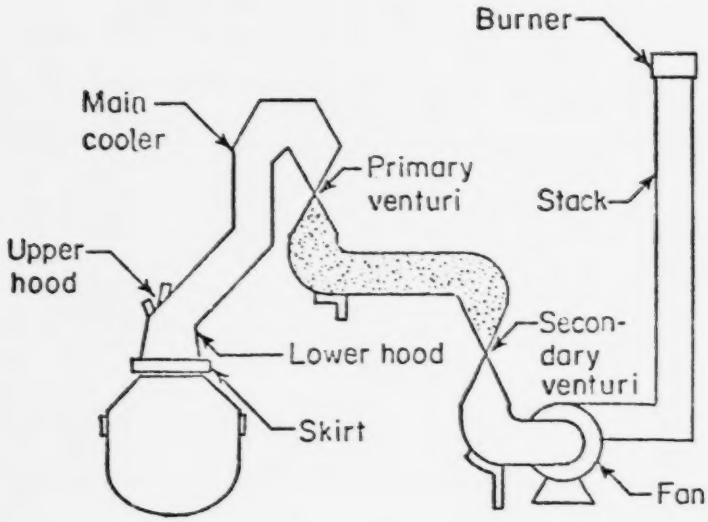


Figure 1. Wet scrubber gas cleaning system

Sampling was performed in 7.62-cm (3-in.) ports on a platform which is located at the 18.3-m (60-ft) level of the 1.83-m (6-ft) diameter by 70.1-m (230-ft) stack. Access to the sampling area is by way of a ladder on the stack.

The velocity pressure and temperature profile of the stack at the sampling location are given in Figure 2. The velocity pressure changed significantly during the course of the 20 minute oxygen blow as may be noted by a typical pattern given in Table 2.

TABLE 2. VELOCITY PRESSURE AND TEMPERATURE PROFILE DURING BLOW - BOF WITH WET SCRUBBER

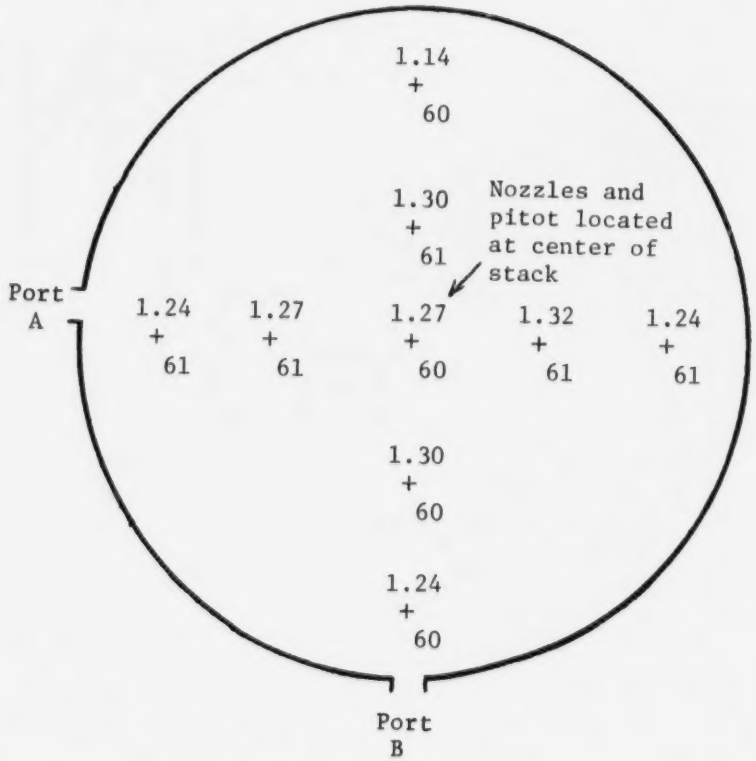
Time into Blow, minutes	Stack Gas Temp., °C	ΔP, cm H_2O
0	54	0.43
2	54	0.89
4	54	0.43
6	56	0.43
8	60	1.14
10	60	1.22
12	58	1.14
14	59	1.14
16	57	0.97
18	59	0.89
20	59	1.78

Sampling Site--ESP Control

Sampling at the site equipped with electrostatic precipitators (ESP) was performed at a shop with two 180-ton furnaces. During sampling only one furnace was in operation; however, the furnaces are frequently operated in tandem. The vessels are charged with about 45 tons of scrap, without preheating, and 135 tons of hot metal. Several low alloy steels are made at the facility through additions to the ladle during teeming. Only molybdenum additions were made directly to the vessel.

During the blow, emissions are collected by an open hood above the vessel and conducted through a cooling section (water spray), then into electrostatic precipitators to remove particulates and finally out through a 5.79-m (19-ft) diameter by 50-m (165-ft) high stack. A diagram of the system is shown in Figure 3.

The general characteristics of the stack emissions from the BOF with ESP are as follows:



Stack diameter - 1.83 m (6 ft)

Reading taken at ∿0.3 m (1 ft) spacings

Upper values are velocity pressure, cm H_2O

Lower values are temperature, °C

Figure 2. Velocity pressure and gas temperature profile of BOF/wet scrubber stack at sampling location

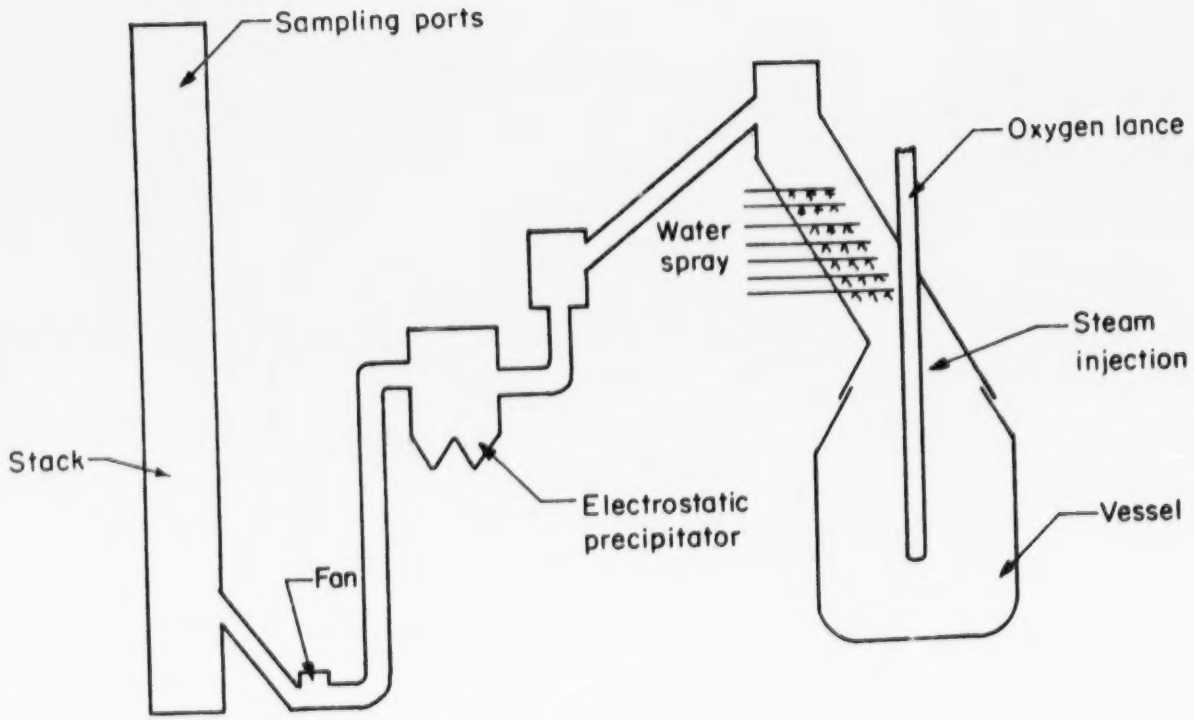


Figure 3. Basic oxygen furnace with electrostatic precipitator emission control

Composition

CO - 0.5 percent
CO_2 - 13 percent
O_2 - 13 percent
NO_x - 176 ppm max
SO_2 - 4 ppm max
Moisture - 16 to 25 percent
Particulates - 30 to 180 mg/m^3

Velocity

~8 m/s

Temperature

130 to 160°C

Sampling was performed at the 46-m (150-ft) level of the stack. The velocity head (ΔP) and temperature profile in the quadrant of the stack in which sampling was performed is shown in Figure 4. Slight variations in ΔP were noted during the first three to five minutes of each oxygen blow and then the ΔP remained steady. The gas temperature changed steadily during the blow, usually increasing by about 25 degrees centigrade from the starting temperature.

SAMPLING EQUIPMENT

Particulates

The particulate sampling was performed with two identical, commercially built, Method 5 trains comprised of components assembled as shown schematically in Figure 5. The sampling probes, which were constructed at Battelle, were 2.06 m (6.8 ft) in length and were constructed from approximately 11-mm-I.D. x 16-mm-O.D. glass tubing. Heating was provided by a 3.05-m (10 ft) glass fiber-insulated heating tape wrapped around the glass probe. A thermocouple junction was taped to the outer surface of the glass tubing at a point between the heater windings and at the midpoint of the probe. The glass probe and heating tape assembly was insulated by a wrapping of asbestos tape. A stainless steel sheath was used to protect the glass probe and to hold the fitting to attach the nozzle. The seal between the glass probe and the 1.6-cm (5/18-in.) Swagelok® nozzle connection was made with a silicone O-ring.

A type "S" pitot tube attached to one of the sampling probes was used for velocity head readings. The tube was constructed at Battelle from approximately 7.5-mm-I.D. x 9.5-mm-O.D. stainless steel tubing. A stainless steel sheathed thermocouple was attached to the pitot tube to provide stack gas temperature measurements. The type "S" pitot tube was calibrated against a standard pitot tube over the velocity range of 10.7 to 29.6 m/sec (35 to 96 ft/sec) in the Battelle wind tunnel facility.

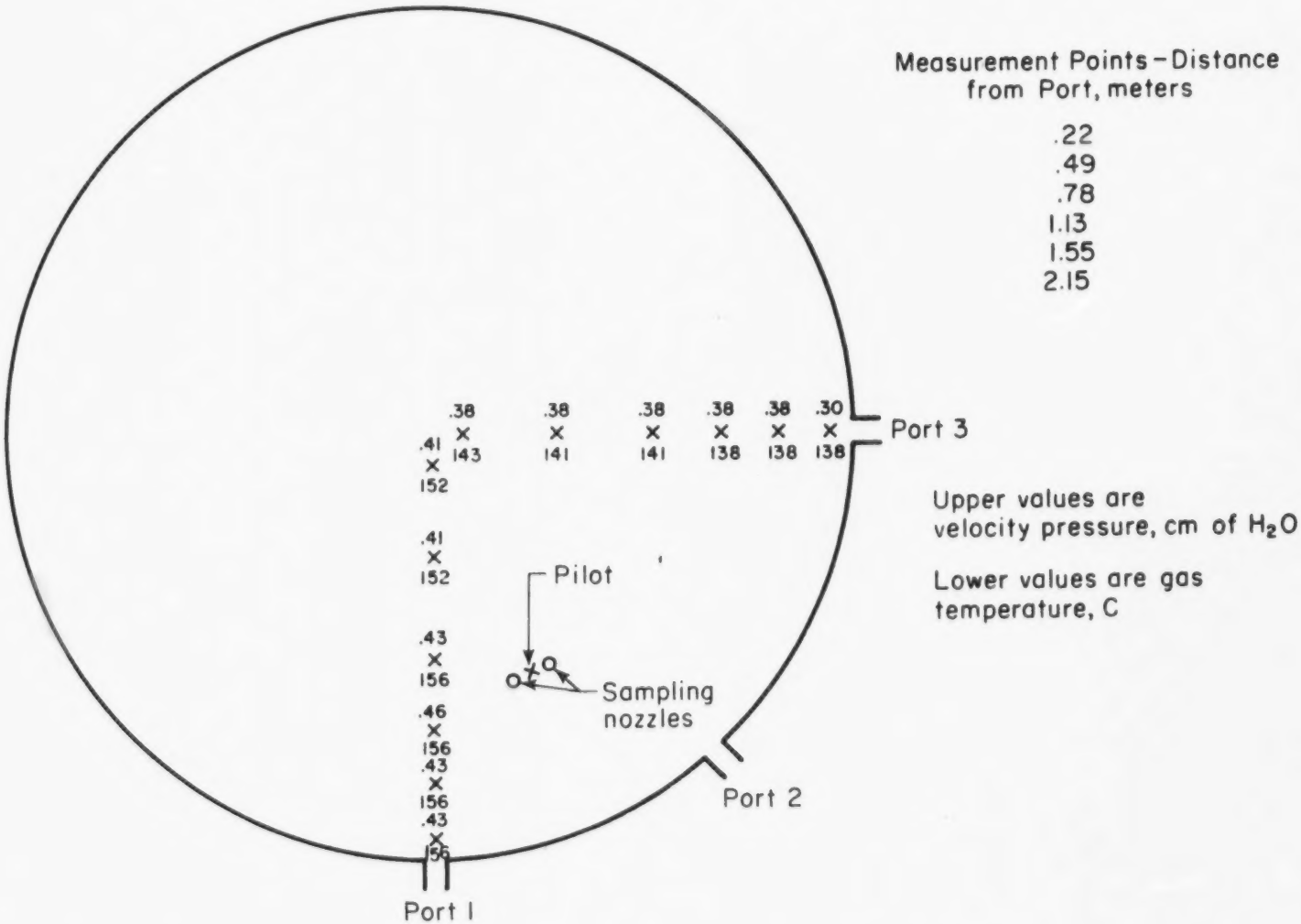
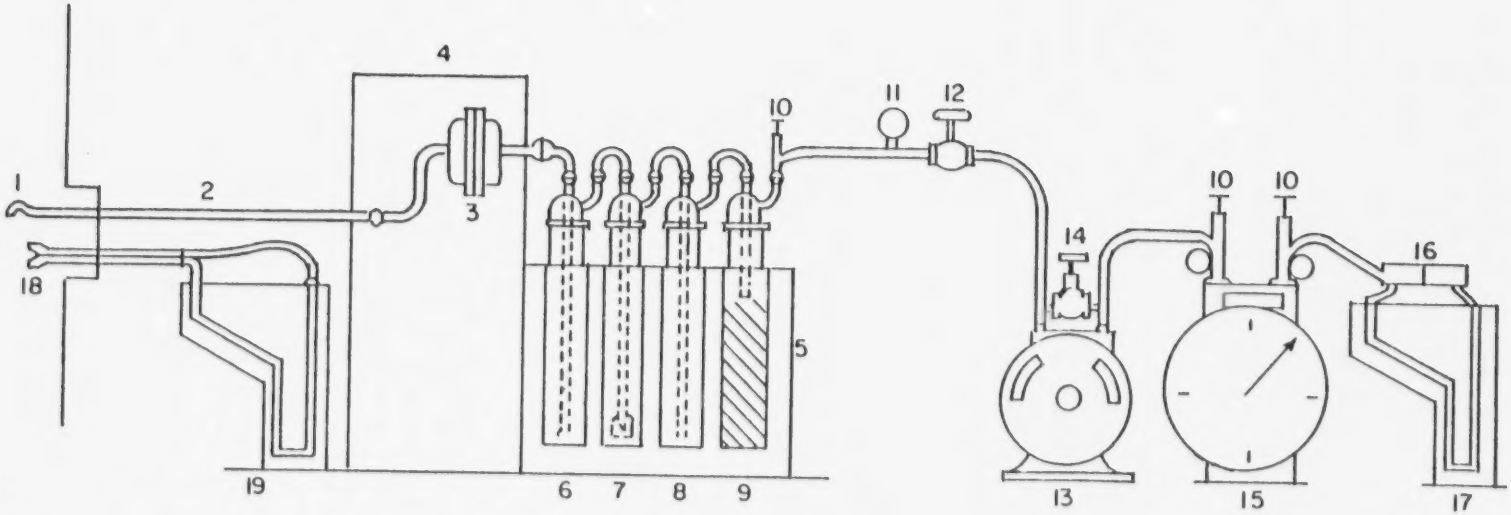


Figure 4. Velocity pressure and gas temperature profile of BOF/ESP stack quadrant at sampling location



1. Stainless steel nozzle
2. Glass-lined probe
3. 7.62 cm (3 in.) filter
4. Heated box for filter
5. Ice bath for impinger
6. Modified G-S impinger with 100 ml water
7. Greenburg-Smith impinger with 100 ml water
8. Modified G-S impinger
9. Silica gel trap
10. Thermometers or thermocouples
11. Vacuum gauge
12. Flow control valve
13. Pump
14. Flow control valve
15. Dry test meter
16. Calibrated orifice
17. Manometer (ΔH)
18. "S" type pitot tube
19. Manometer (ΔP)

Figure 5. Schematic diagram of Method 5 train

Two modifications were made in the Method 5 sampling trains to permit additional gas temperature measurements at various points and to control the gas outlet probe temperature. The glass connectors from the probe outlet to the filter and the filter outlet to the first impinger were modified for some experiments as shown in Figures 6 and 7. The probe-to-filter connector contained a thin-wall thermocouple well which extended about 5.1 cm (2 in.) into the outlet end of the probe. The modified filter-to-first impinger connector was fitted with a metal thermometer, the tip of which was positioned about 1.3 cm (0.5 in.) from the frit in the filterholder.

In-Stack Filters--

In-stack sampling was performed at the BOF/wet scrubber using a Gelman #2220, 47-mm filterholder. Due to the entrained moisture in the emissions, it was necessary to heat the filterholder to maintain a dry filter during sampling. Heating was provided by a silicone rubber-insulated heating tape wrapped around the filterholder. A metal case was fitted around the entire assembly to protect it from the moisture. A thermocouple was attached to the outside of the filter case to measure and regulate temperature.

Two types of in-stack filter configurations were used in sampling at the ESP equipped BOF facility. The one type was a 6.25-cm (2.5 in.) filterholder marketed by Sierra Instrument Company Model 8145. The other type of in-stack filterholder, shown in Figure 8, was constructed at Battelle and was designed to use Munktell glass fiber thimbles* which are marketed by Carborundum Company. The thimble was sealed to the inlet with the spring loaded, stainless steel collar. A Teflon® gasket was used to seal the filter-holder body.

The in-stack filterholders were inserted between the nozzle and the glass-lined probe. All other components of the sampling train were assembled as shown in Figure 5.

Gas Sampling

Continuous monitoring for SO_2 and NO_x was performed at both BOF facilities with an Environmetrics Model NS 300AC Faristor unit. The gas sample was extracted from the stacks through a 0.63-cm (0.25-in.) diameter stainless steel tube and passed through a moisture trap prior to the Faristor unit. Teflon® lines were used to connect the monitor to the sampling probe.

Evaculated three liter flasks were used at the BOF/ESP facility to collect samples for NO_x and gas mass spectrometric analysis. CO, CO_2, and O_2 were determined with Orsat and Fyrite equipment.

*Munktell's Swedish glass fiber thimbles are made by Grychksbo Papersbruk, AB, Sweden.

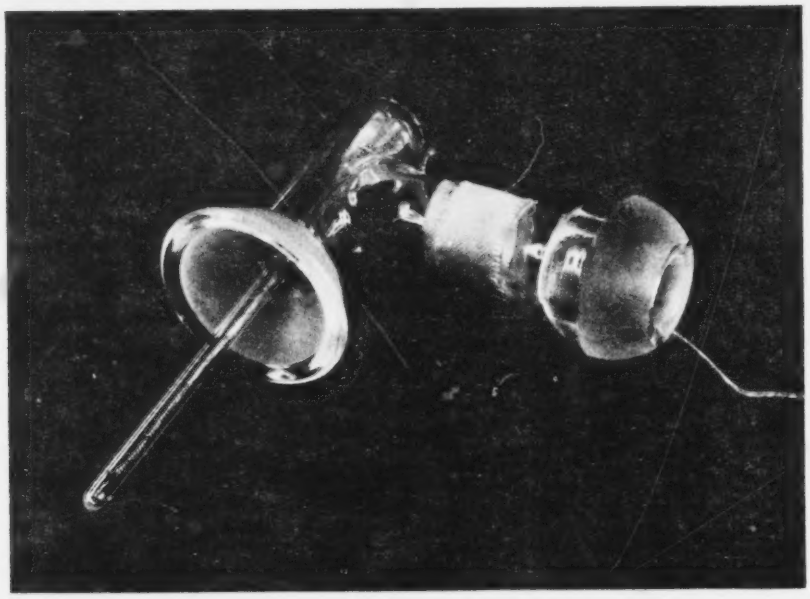


Figure 6. Thermocouple assembly used to measure gas temperature at probe outlet

Figure 7. Thermometer assembly used to measure gas temperature at the box filter outlet

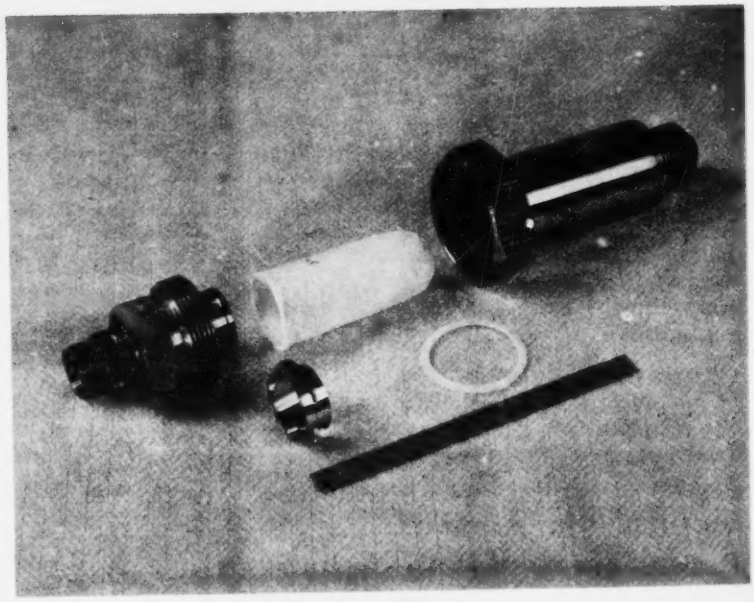


Figure 8. In-stack filterholder for glass fiber thimbles

SAMPLE COLLECTION AND ANALYSIS PROCEDURES

Particulate Sampling

In all tests, particulate sampling was performed concurrently with two systems (designated A and B), each with a separate operator. The sampling was performed at a fixed-point in the stack in an area of nearly uniform velocity. Sampling probes of the two systems were inserted into the duct through two adjacent ports so that the pitot tube attached to one of the probes was positioned equidistance between the sampling nozzles. The separation between the pitot tube and each nozzle was about 2.5 cm (1 in.). The relative nozzle-pitot tube positions and the point of sampling within the duct are indicated in Figures 2 and 4.

At the start of each test day, the laboratory calibration of the gas metering components of both sampling systems was checked by setting the orifice manometer (ΔH) to the meter box calibration factor ($\Delta H_@$) and measuring the flow rate through the dry gas meter over a 5-minute period. A flow rate of 0.21 m^3/min (0.75 cfm) confirmed that the gas metering system remained in calibration.

The preparation of the particulate collection trains for all tests was performed as specified in Paragraph 4.1.2 of Method 5 with the following modifications:

(1) The entire sampling train was leak checked by plugging the sampling nozzle inlet and evacuating to 38.1 cm (15 in.) of Hg. Leak rates did not exceed 566 cm^3/min (0.02 cfm). This procedure was used for both the regular Method 5 train and the in-stack filter train. Prior to leak testing, the in-stack filter assembly was heated to stack temperature with a heat gun. Heating was continued until the probe was inserted into the duct to initiate sampling.

(2) The probe was heated until the thermocouple at the outlet indicated that the desired operating temperature was achieved prior to initiation of sampling.

(3) Heat guns (260-399°C) were necessary on some occasions for supplemental heating when the box filter was operated at temperatures above 121°C (250°F).

In performance of the experiments, sampling trains were operated as prescribed in Paragraph 4.1.3 of Method 5. The ΔP, ΔH, and system temperatures were read each minute during the first 5 minutes of the blow and at 5-minute intervals thereafter. Frequent adjustment of sampling rate was required to maintain isokinetic conditions during the initial minutes of sampling. The stack velocity was determined by only one pitot tube. However, two nomographs were used to obtain the proper sampling rate (ΔH). Temperature measurements were obtained at the points shown in Figure 9.

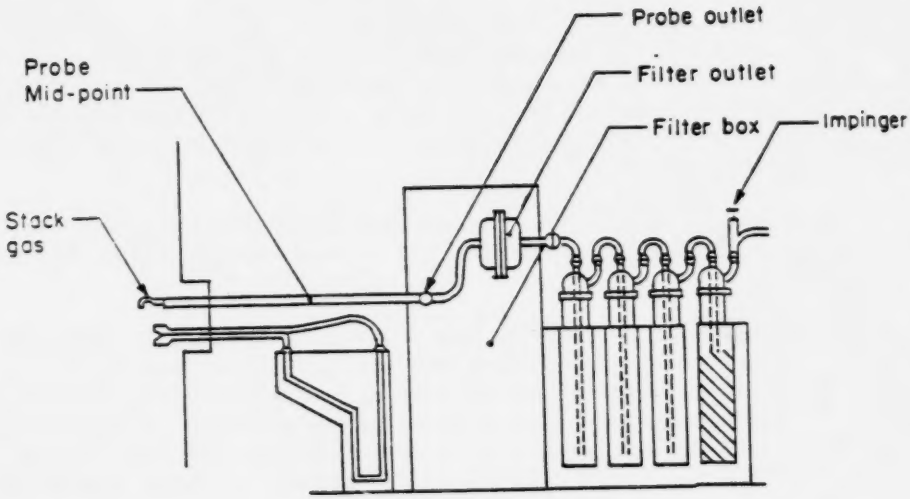


Figure 9. Temperature measurement points

Sampling periods were initiated after the hot metal charge and continued until the end of the oxygen blow. Each test consisted of sampling during three or four blows.

After completion of the tests, the trains were again leak checked, sealed to prevent contamination, and transferred to the sample recovery area.

Sample Recovery and Analysis

Filters were removed from holders, sealed in Petri dishes or glass jars (thimbles), and immediately placed in a desiccator. In Method 5-type tests, the probe and nozzle were disassembled and washed separately. The probe was first rinsed with acetone without brushing, then rinsed with acetone while slowly inserting and removing a Nylon® brush in a rotating fashion. The acetone wash and brushing were continued until visual inspection indicated that all particulates were removed. The brush was thoroughly flushed with acetone prior to removal from the probe. The probe wash (usually about 100 to 150 ml) was collected in an Erlenmeyer flask sealed onto the probe outlet ball joint. Particulates were recovered from the nozzle and the inlet half of the filter holder by alternately brushing and rinsing with acetone. The wash solutions from all three components (probe, nozzle, and filterholder) were combined for analysis.

In all tests with in-stack filters, the in-stack filter was removed from the probe and particulates were recovered from the probe as described in the previous paragraph. The outlet side of the in-stack filterholder and the inlet side of the back-up filterholder (box filter) were separately brushed and washed with acetone and the solutions were combined with the probe wash. The nozzle and inlet side of the in-stack filterholder, as one unit, was alternately brushed and washed with acetone. This wash solution was not combined with the probe wash, but analyzed separately.

At least one 200-ml acetone blank was obtained each day from the wash bottle dispenser. All acetone wash solutions and blanks were stored in glass bottles with Teflon®-lined caps for transfer to the laboratory for analysis.

The MSA 1106 BH and ADL quartz filters and particulate catches were desiccated at least 24 hours (usually longer) prior to weighing. It was found necessary to desiccate the Munktell (Carborundum) thimbles at least 72 hours prior to weighing (both tare and final) to achieve a constant weight.

The acetone wash solutions were evaporated to dryness in a reverse airflow, clean hood and the residues were desiccated to a constant weight to the nearest 0.1 mg.

Calculations were performed as described in Section 6 of Method 5.

Gas Analyses

Continuous analyses for SO_2 and NO_x were performed concurrently with all tests in the preliminary series of particulate sampling experiments.

A gas sample was withdrawn from the BOF stack emissions at the rate of about 1.5 liters/minute passed through an ice-cooled moisture trap and analyzed with an Environmetrics NS300 AC monitor. Calibrations were performed at the start and end of each test day by passing standard gas mixtures through the sample inlet system. Grab samples for mass spectrometric analysis were taken with evacuated 2-liter glass flasks. Orsat analysis were made on integrated bag samples (Method 3) collected over the oxygen blowing period.

TEST DESCRIPTIONS AND RESULTS

BOF with Wet Scrubber Emission Control

Experiments were performed to compare in-stack sampling with Method 5 and to evaluate the sensitivity of Method 5 to the operating variables--system temperature and deviation from isokinetic sampling rate. A summary of the experimental conditions is given in Table 3. Runs 1 through 13 were conducted to study the characteristics of in-stack sample collections and to compare this technique with Method 5. Two different filter materials, Pallflex Tissuquartz 2500 QAO and MSA 1106BH, were used in the Method 5 trains to study filter/emission interactions.

The results of the BOF/wet scrubber experiments involving comparison of in-stack and Method 5 sampling are presented in Table 4. Sampling and stack gas data from these experiments are given in Appendices B and C.

In Runs 1 through 4, it was impossible to heat the in-stack filter sufficiently to prevent moisture accumulation. A high pressure drop across the in-stack filter resulted which caused rupture of the filter (Run 4) or inability to sample at an isokinetic rate. The comparatively higher loadings noted in Runs 1A, 2A, and 3A are probably a consequence of subisokinetic sampling.

In the subsequent experiments, the in-stack filterholder, wrapped with a heating tape, was tightly sealed into a metal enclosure. This arrangement apparently succeeded in preventing condensation or accumulation of entrained water droplets on the in-stack filters.

The comparison of the mass loading results of the valid in-stack--Method 5 tests (Runs 5 through 13) shows considerable scatter in the data. Most notable is the fact that for some unknown reason the Method 5 train with MSA 1106BH filters gave considerably higher results than in-stack sampling in Runs 12 and 13. However, two other runs (5 and 11) with the same filter material gave in-stack and Method 5 results which were in much better agreement.

In general, in-stack and Method 5 comparisons using the same filter medium, Tissuquartz, showed higher mass loadings for the in-stack sampling train. Based on the average of the four runs (6, 8, 9, and 10), the in-stack results (filter and nozzle only) are 12 percent higher than the Method 5 values.

TABLE 3. SUMMARY OF EXPERIMENTS -- BOF WITH WET SCRUBBER

Run #	Filter Configuration	Filter Material	Other variables	Run #	Filter Configuration	Filter Material	Other variables
1A	In-stack	Tissuquartz		10A	In-stack	Tissuquartz	
B	Method 5	Tissuquartz		B	Method 5	Tissuquartz	
2A	In-stack	Tissuquartz		11A	In-stack	Tissuquartz	
B	Method 5	Tissuquartz		B	Method 5	MSA 1106	
3A	In-stack	Tissuquartz		12A	In-stack	Tissuquartz	
B	Method 5	Tissuquartz		B	Method 5	MSA 1106	
4A	In-stack	Tissuquartz		13A	In-stack	Tissuquartz	
B	Method 5	MSA 1106		B	Method 5	MSA 1106	
5A	In-stack	Tissuquartz		14A	Method 5	MSA 1106	System Temperature ∿121C
B	Method 5	MSA 1106		B	Method 5	MSA 1106	System Temperature ∿191C
6A	In-stack	Tissuquartz		15A	Method 5	MSA 1106	System Temperature ∿121C
B	Method 5	Tissuquartz		B	Method 5	MSA 1106	System Temperature ∿149C
7A	In-stack	Tissuquartz		16A	Method 5	MSA 1106	System Temperature ∿121C
B	Method 5	Tissuquartz		B	Method 5	MSA 1106	System Temperature ∿121C
8A	In-stack	Tissuquartz		17A	Method 5	MSA 1106	∿100% isokinetic
B	Method 5	Tissuquartz		B	Method 5	MSA 1106	∿130% isokinetic
9A	In-stack	Tissuquartz		18A	Method 5	MSA 1106	∿70% isokinetic
B	Method 5	Tissuquartz		B	Method 5	MSA 1106	∿100% isokinetic
				19A	Method 5	MSA 1106	System Temperature ∿121C
				B	Method 5	MSA 1106	System Temperature ∿71 C

TABLE 4. SAMPLE WEIGHT DATA

Run No.	Sample Weights, mg: In-Stack Filter	Box Filter	Probe	Nozzle	Probe/ Nozzle	Total[a]	mg/Nm³	Ratio $\frac{mg/Nm^3\text{-A}}{mg/Nm^3\text{-B}}$[a]	Distribution in Sampling System, % Total Catch: In-Stack Filter	Box Filter	Probe	Nozzle	Probe/ Nozzle
1A	46.3	0.0	8.6	8.7	--	63.6 (55.0)	62.0 (53.6)	1.56	72.8	0.0	13.5	13.7	--
1B	--	31.1	--	--	18.9	50.0	39.8		--	62.2	--	--	37.8
2A	93.4[b]	33.4[b]	181.7	31.3	--	339.8	130.4	--[b]	--	--	--	--	--
2B	--	100.0	--	--	66.3	166.3	34.7		--	60.1	--	--	39.1
3A	64.9[b]	39.8[b]	87.5	16.9	--	209.1	100.5	--[b]	--	--	--	--	--
3B	--	119.7	--	--	34.1	153.8	33.3		--	77.8	--	--	22.2
4A	12.9	32.3	10.6	8.5	--	64.3	36.1	--[c]	--	--	--	--	--
4B	--	57.4	--	--	10.0	67.4	28.5		--	85.2	--	--	14.8
5A	42.4	0.0	4.5	4.6	--	51.5 (47.0)	24.9 (22.7)	1.00 (0.91)	82.4	0.0	8.7	8.9	--
5B	--	61.7	--	--	4.7	66.5	24.9		--	92.9	--	--	7.1
6A	38.1	0.1	8.0	4.4	--	50.6 (42.5)	30.5 (25.6)	1.18 (0.99)	75.4	0.2	15.7	8.6	--
6B	--	40.5	--	--	4.4	44.9	25.8		--	90.2	--	--	9.8
7A	14.8[c]	34.5	5.6	1.8	--	56.7	33.1	--[c]	--	--	--	--	--
7B	--	50.4	--	--	8.1	58.5	32.8		--	86.2	--	--	13.8
8A	29.8	0.0	4.4	9.5	--	43.7 (39.3)	69.4 (62.4)	1.26 (1.13)	68.1	0.0	10.1	21.8	--
8B	--	15.1	--	--	32.2	47.3	55.1		--	31.8	--	--	68.2
9A	39.7	1.4	5.8	6.2	--	53.1 (45.9)	37.2 (32.2)	1.30 (1.12)	74.7	2.7	10.9	11.6	--
9B	--	24.9	--	--	18.0	42.9	28.7		--	58.0	--	--	42.0
10A	54.5	0.0	2.2	11.6	--	68.3 (66.1)	43.7 (42.3)	1.27 (1.23)	79.8	0.0	3.2	17.0	--
10B	--	26.5	--	--	29.0	55.5	34.4		--	47.7	--	--	52.3
11A	64.9	0.0	6.2	4.7	--	75.8 (69.6)	38.4 (35.3)	1.20 (1.10)	85.6	0.0	8.2	6.2	--
11B	--	29.6	--	--	37.3	66.9	32.1		--	44.2	--	--	55.8
12A	41.0	0.8	4.1	8.0	--	53.9 (49.0)	32.1 (29.2)	0.64 (0.58)	76.1	1.5	7.5	14.9	--
12B	--	32.6	--	--	53.2	85.8	50.0		--	38.0	--	--	62.0
13A	32.6	0.0	2.8	8.3	--	43.7 (40.9)	27.6 (25.8)	0.78 (0.73)	74.6	0.0	6.5	18.9	--
13B	--	22.9	--	--	35.1	58.0	35.4		--	39.5	--	--	60.5
								Averages - System A	76.7	0.4	9.4	13.5	--
								Averages - System B	--	62.6	--	--	37.3

(a) Data based on total system catch. Values in parenthesis are calculated from filter and nozzle collections only.
(b) In-stack filter appeared that moisture had condensed on it sometime during test.
(c) In-stack filter ruptured.

The operating temperature of the in-stack filter was difficult to maintain at a constant level. Average reading for Runs 5 through 13 ranged from 113°F to 164°F. However, the comparative mass loading results (A/B) show no correlation with in-stack temperature variations.

The results of experiments with Method 5 to evaluate the effects of anisokinetic sampling and system temperature are given in Table 5. Stack gas and sampling data are given in the appendices.

In Runs 17 and 18 the sampling was performed by a pair of Method 5 trains, one of which was operated under anisokinetic conditions. The comparison of the results indicate that deviations of 1.3 and 0.7 from isokinetic sampling rate do not introduce appreciable error in the Method 5 mass measurement.

In the other experiments sampling was performed with a pair of Method 5 trains, one of which was operated at the minimum specified gas temperature at the probe outlet, i.e., 121°C. The filter box on this system was also held at 121°C (250°F). The gas temperature at the probe outlet and the filter box temperature of the other Method 5 trains was maintained at 191°C (375°F) for Run 14, 149°C (300°F) for Run 15, and at about stack temperature, ∿84°C (183°F) for Run 19. The mass results obtained with the systems in which temperature was varied above and below 121°C (250°F) exhibit excellent agreement with the "normal" Method 5 data. The variations are not significantly different from those observed in the two concurrent Method 5 measurements (Run 16).

If it is assumed that there is no interaction of system temperature with the mass results, Runs 14, 15, 16, and 19 may be used to obtain an estimate of Method 5 precision. Based on these data, repeatability (within-laboratory precision) of Method 5 for sampling the wet scrubber equipped BOF is estimated to be 2.8 percent.

BOF with ESP Emission Control

The experiments at the BOF/ESP facility included a series of experiments of confounded factorial design[8] to study the effects of filter media and sampling system temperature on the mass results and a test series to compare Method 5 and two types of in-stack filter configurations.

The effect of Method 5 sampling temperature (filter box and gas at probe outlet) was studied at two levels: the minimum specified temperature, 121°C (250°F) and at approximately the temperature of the BOF stack gas, 177°C (350°F). Two filter materials, MSA 1106BH and an ADL-developed high purity quartz fiber, were included in the experimental design. The pattern for the filter media--sampling system temperature experiments is presented in Table 6 and results of the experiments are given in Table 7. Statistical analysis of the data was performed by analyses of variance techniques[8] yielding the statistical data and conclusions given in Table 8. Based on the criteria of a 95 percent confidence level, it is concluded that differences in results obtained with sampling system temperatures of 121°C

TABLE 5. SAMPLE WEIGHT AND MASS LOADING DATA - BOF WITH WET SCRUBBER

Run No.		Sample Weights, mg			mg/m^3
		Filter	Probe	Total	
14A	System ∿121°C	85.3	34.3	119.6	56.3
14B	System ∿191°C	85.5	24.0	110.5	53.1
15A	System ∿121°C	43.7	14.6	58.3	33.4
15B	System ∿149°C	43.1	13.3	56.4	34.0
16A	System ∿121°C	79.2	37.4	116.6	61.9
16B	System ∿121°C	76.8	33.3	110.1	59.9
17A	∿100% Isokinetic	42.6	13.7	56.3	33.3
17B	∿130% Isokinetic	52.0	25.1	77.1	36.2
18A	∿70% Isokinetic	51.8	12.0	63.8	46.8
18B	∿100% Isokinetic	69.7	14.7	84.4	46.1
19A[a]	System ∿121°C	18.1	6.6	24.7	54.2
19B[a]	System - stack temp.	17.4	6.2	23.6	55.4

(a) Run included only one blow. Process interruption precluded additional sampling.

TABLE 6. RANDOMIZED TEST PATTERN FOR STUDY OF FILTER MEDIA, TEMPERATURE, AND THEIR INTERACTION - (BOF WITH ESP EMISSION CONTROL)

Rep	Block	Test Number	System A			System B		
			Temp., °C	Filter	Operator	Temp., °C	Filter	Operator
1	1	1	∿177	ADL	1	121	MSA	2
	2	2	121	ADL	2	∿177	MSA	1
2	1	3	121	ADL	2	∿177	ADL	1
	2	4	∿177	MSA	2	121	MSA	1
3	1	5	121	MSA	1	121	ADL	2
	2	6	∿177	ADL	1	∿177	MSA	2

This design represents a confounded factorial design, with the following confounding:

Tests 1 and 2 confound FxT interaction with blocks.
Tests 3 and 4 confound F main effect with blocks.
Tests 5 and 6 confound T main effect with blocks.

The analysis of variance will yield 2/3 information on each factor.

TABLE 7. SAMPLE WEIGHT DATA - TEMPERATURE/FILTER EXPERIMENTS - BOF WITH ESP EMISSION CONTROL

Run No.	System Temperalture, °C[a]		Filter Material	Sample weights, mgs			mg/Nm^3
	Box Filer	Probe Outlet		Filter	Probe	Total	
1A	176	179	ADL	43.2	18.9	62.1	33.7
1B	122	122	MSA	44.2	17.7	61.9	34.1
2A	121	129	ADL	36.8	21.2	58.0	32.8
2B	177	178	MSA	50.7	20.4	71.7	40.5
3A	123	123	ADL	199.1	108.4	307.5	165.4
3B	177	179	ADL	229.4	94.0	323.4	177.4
4A	177	176	MSA	123.7	34.5	158.2	118.2
4B	120	128	MSA	93.5	64.6	158.1	120.4
5A	126	127	MSA	154.2	47.4	201.6	139.1
5B	122	127	ADL	137.0	55.2	192.2	138.4
6A	179	179	ADL	71.7	34.3	106.0	92.6
6B	177	185	MSA	70.8	27.1	97.9	85.4

(a) Actual average system temperatures.

TABLE 8. ANALYSES OF VARIANCE-TEMPERATURE/FILTER EXPERIMENTS--
BOF WITH ESP EMISSION CONTROL

Source	Degrees of Freedom	Sum of Squares	Mean Square	F-Ratio[a]	Conclusion	Blocks with Information
Filter (F)	1	0.32	0.32	0.03	Not significant	1, 2, 5, 6
Temp. (T)	1	36.55	36.55	3.39	Not significant	1, 2, 3, 4
FxT	1	61.05	61.05	5.66	Not significant for $\alpha \leq 0.05$ Significant for $\alpha = 0.10$	3, 4, 5, 6
Reps	2	25,713.52	12,856.76			All
Block/Reps	3	5,197.04	1,732.35			All
Remainder	3	32.39	10.80			All
Total	11	31,040.87	2,821.90			All

(a) Critical values of F-Ratio.

α	Critical Value
0.100	5.54
0.050	10.13
0.025	17.44
0.010	34.12

(250°F) and 177 (350°F) and with the MSA 1106BH and the ADL developed quartz fiber filter materials are not statistically significant. Furthermore, there is no statistically significant interaction effect between sampling system temperature and filter media.

Assuming no filter or temperature effects, results of Runs 1 through 6 may be used to estimate Method 5 precision. Based on these data, the repeatability (within-laboratory precision) expressed as the coefficient of variation is 3.4 percent.

Four experiments (Runs 7 through 10) were performed to compare results of Method 5 and in-stack sampling techniques. In two experiments, in-stack glass fiber thimbles marketed by Carborundum Company were used and in the others, 6.25 cm (2.5 in.) MSA 1106BH in an in-stack flat filterholder were employed. MSA 1106BH filter material was used in the Method 5 trains. The results of the experiments are presented in Table 9. Both the in-stack thimble and flat filter based on the filter and nozzle catches gave lower mass loading results than the corresponding Method 5 values. With exception of Run 7, mass loadings were also lower when calculated from the total in-stack system particulate collection. Based on nozzle and filter catches, the in-stack flat and thimble filters gave results which were an average of 25 and 16 percent lower, respectively, than Method 5 results.

Statistical analysis of the test pairs does not indicate that the differences between the in-stack and Method 5 results are significant. However, the power of the statistical analysis is greatly diminished by the experimental design and number of replications. Statistical conclusions aside, the consistency of the in-stack and Method 5 differences would seem to argue that the two methods do not give equivalent results.

CHARACTERIZATION OF PARTICULATE COLLECTIONS AND GASEOUS EMISSIONS

Chemical Composition of Particulates

Extensive chemical analyses of particulate and gas samples from both BOF facilities were conducted to investigate possible interactions in the sampling process and to determine if the particulate collections were representative of the stack emissions. Table 10 presents the results of optical emission spectroscopy analysis of Method 5 filter and probe catches and samples taken from the wet scrubber emission control system. In general, the compositions of the Method 5 filter collections are essentially the same as particulates removed by the wet scrubber (clarifier sludge). Comparison of the filter and probe collections shows a disproportionate distribution of several elements, e.g., Fe, K, and Na. The relatively higher Na and K concentrations in the probe may arise from entrained liquid droplets in the stack emissions. Analysis indicates that these elements comprise the major fraction of the dissolved solids in the scrubber liquid.

Compositional analysis of particulates collected from the BOF/wet scrubber facility by Method 5 and in-stack sampling are given in Table 11.

TABLE 9. SAMPLE WEIGHT AND DISTRIBUTION DATA - METHOD 5 AND IN-STACK COMPARISON TESTS - BOF WITH ESP EMISSION CONTROL

Run No.	Sampling[a] Method	Sample Weights, mgs					
		Box Filter	In-stack Filter	Probe	Nozzle	Total	mg/Nm^3[b]
7A	Method 5	115.1	--	46.7[c]	--	161.8	131.6
7B	In-stack thimble	0.9	138.7	16.3	10.1	166.0	121.1 (135.2)
8A	Method 5	74.6	--	37.4[c]	--	112.0	93.4
8B	In-stack thimble	0.0	77.5	11.9	9.0	98.4	71.6 (81.5)
9A	Method 5	60.5	--	45.2[c]	--	105.7	93.1
9B	In-stack flat	0.9	69.1	15.5	7.5	93.0	68.3 (82.9)
10A	Method 5	67.3	--	41.0[c]	--	108.3	86.4
10B	In-stack flat	1.2	73.6	17.3	8.5	100.6	66.4 (81.4)

Run No.	Sampling[a] Method	Sample Distribution, Percent of Total Collection					
		Box Filter	In-Stack Filter	Probe	Nozzle	In-Stack Filter and Nozzle	Behind In-Stack Filter
7A	Method 5	71.1	--	28.9[c]	--	--	--
7B	In-stack thimble	0.5	83.6	9.8	6.1	89.6	10.4
8A	Method 5	66.6	--	33.4[c]	--	--	--
8B	In-stack thimble	0.0	78.8	12.1	9.1	87.9	12.1
9A	Method 5	57.2	--	42.8[c]	--	--	--
9B	In-stack flat	1.0	74.3	16.7	8.1	82.4	17.6
10A	Method 5	62.1	--	37.9[c]	--	--	--
10B	In-stack flat	1.2	73.2	17.2	8.4	81.6	18.4

(a) MSA 1106BH filters were used for Method 5 and in-stack flat filter experiments. In-stack thimble was Munktell glass fiber filter.

(b) Based on nozzle and in-stack filter catch. Values in parenthesis are calculated from total system catch.

(c) Probe and nozzle rinse combined.

TABLE 10. ANALYSIS OF METHOD 5 AND SCRUBBER SAMPLES -- BOF WITH WET SCRUBBER EMISSION CONTROL

Sample	Weight Percent in Sample[a]																			
	Fe	Si	Na	K	Ca	Zn	Mg	Mn	Al	Pb	B	Ti	Sn	Ni	Co	Cr	V	Mo	Cu	Ag
Method 5 Filter Run 14A	30-50	1	1	2-3	0.5	1	0.1	0.5	0.02	0.5	0.01	<0.005	--	0.003	0.003	0.01	0.005	0.003	0.01	<0.001
Method 5 Filter Run 16A	30-50	1	1	2-3	2	5-10	0.3	0.5	0.02	1	0.01	0.005	--	0.003	0.003	0.01	0.01	0.003	0.03	0.001
Method 5 Probe Residue Run 14A	15-30	1	4-7	10-20	1	2-3	0.3	0.5	0.3	0.5	0.02	0.01	--	0.03	<0.003	0.03	<0.005	0.003	0.03	0.005
Method 5 Probe Residue Run 16A	15-30	1	4-7	10-20	1	2-3	0.3	0.5	0.3	0.5	0.03	0.02	--	0.03	<0.003	0.01	<0.005	0.003	0.03	0.003
Clarifier sludge[b]	40-60	0.5	<0.1	<0.1	2	2-3	0.3	0.6	0.03	0.3	<0.01	0.01	<0.01	0.003	<0.01	0.01	0.005	0.003	0.03	0.001
Suspended solids[b]	40-60	0.5	0.1	<0.1	2	0.2	0.6	2	0.03	0.03	<0.01	0.005	<0.01	0.005	<0.01	0.01	--	--	--	--
Dissolved solids[b]	0.01	0.1	30-50	30-50	0.3	<1.0	0.2	<0.1	0.01	<0.01	0.01	<0.005	<0.01	<0.005	<0.01	<0.01	--	--	--	--

(a) Analyses performed by optical emission spectroscopy.

(b) Samples of stack particulates taken from scrubber cleanup system.

TABLE 11. CHEMICAL COMPOSITIONS OF PARTICULATE COLLECTIONS FROM BOF WITH WET SCRUBBER
(Results in weight percent)

Run	System/ Component	Fe	Si	Na	K	Mn	Ca	Al	Mg	Zn	Pb	Other Metals	C Uncombined	$CO_3^=$	$SO_4^=$	F^-	Cl^-	Total of Average Individual Determinations (a)
8A	In-stack/	27.5	∿3	9.0	16.0	--	--	∿0.2	∿0.5	--	--	--	1.0	15.2	--	2.1	4.5	
9A	Filter	31.5	∿3	5.0	6.3	1.0	0.9	∿0.5	∿0.5	0.3	0.5	--	--	--	4.0	1.1	--	
11A		25.0	∿3	6.1	14.0	1.2	0.5	∿0.7	∿0.5	1.2	0.4	--	--	--	4.0	1.8	9.0	
12A		38.5	∿3	5.1	10.5	--	--	--	--	--	--	--	0.75	10.0	6.0	2.7	3.0	
13A		40.5	∿3	5.5	16.0	--	--	∿0.2	--	0.6	0.3	--	0.4	16.0	3.5	1.8	--	
Average		32.2	3	6.1	12.6	1.1	0.7	∿0.4	∿0.5	0.7	0.4	<0.1	.7	13.7	4.4	1.9	5.3	∿100%
8B	Method 5/	47.5	∿3	6.0	13.1	--	--	--	--	--	--	--	3.0	10.0	8.0	3.4	5.3	
9B	Filter	55.0	∿3	3.0	9.0	1.7	1.6	∿0.5	∿0.2	0.5	∿0.3	--	--	--	2.1	2.9	2.3	
11B		59.0	∿3	1.0	8.0	2.1	1.0	∿0.5	∿0.2	1.8	∿0.3	--	--	--	4.2	1.5	2.3	
12B		41.0	∿3	6.5	4.5	--	--	--	--	--	--	--	0.2	7.3	3.0	3.5	4.5	
13B		60.0	∿3	7.0	6.5	--	1.9	--	--	--	--	--	0.4	6.5	5.3	4.0	5.0	
Average		51.5	3	4.7	8.2	1.9	1.5	∿0.5	∿0.2	1.2	∿0.3	<0.1	1.2	7.9	4.5	3.1	3.9	117.4%
9A	In-stack/	∿15.	∿3	∿1.	∿2.	∿1.	∿1.	∿1.	∿0.3	0.5	∿0.3	--	--	--	--	--	--	
11A	Nozzle	∿19.	∿3	∿0.5	∿2.	∿2.	∿2.	∿0.3	∿0.5	0.5	∿0.5	--	--	--	--	--	--	
8A, 12A, 13A		26.0	∿3	4.0	5.0	3.0	--	∿0.2	--	--	--	--	12	31.5	6.5	2.5	2.0	
Average		20.0	∿3	1.8	3.0	2.0	1.5	0.5	0.4	0.5	0.4	<0.1	12	31.5	6.5	2.5	2.0	99.5%
9A	In-stack/	6.6	∿3	∿2.	∿2	∿0.7	∿1.	∿1.	∿0.8	∿1.	∿0.2	--	--	--	--	--	--	
11A	Probe	2.4	∿3	∿10.	∿10.0	∿0.4	∿0.9	∿0.5	∿0.5	∿0.3	∿0.2	--	1.0	20.0	13.0	2.5	2.0	
Average		4.5	∿3	∿6.	∿6.	0.5	1.	0.8	0.7	0.7	0.2	<0.1	1.0	20.0	13.0	2.5	2.0	67.2%
8B	Method 5/	13.2	--	7.9	18.5	--	--	--	--	--	--	--	10.	27.9	12.9	2.8	6.2	
9B	Nozzle and	∿11.	∿3	∿10.	∿10	∿1	∿1	∿1.	∿0.5	∿0.2	∿0.5	--	--	--	--	--	--	
11B	Probe	8.7	∿3	8.0	22.9	0.85	0.6	0.5	∿0.2	∿1.	∿0.3	--	0.3	15.0	29.0	2.0	9.0	
12B		7.2	--	10.0	20.0	--	--	--	--	--	--	--	3.0	20.0	18.8	3.2	9.3	
13B		7.4	--	8.7	16.3	1.	1.4	∿0.25	1.	∿0.2	--	--	4.0	25.0	13.8	2.6	8.0	
Average		9.5	3.	8.9	18.0	1.	1.0	0.5	0.5	∿0.4	0.4	--	4.2	22.0	18.6	2.7	8.1	106.2%

(a) Fe and Si calculated as oxides.

In general, the analyses were obtained by use of an optical emission spectrographic method to show the approximate cation content of the samples. These analyses were followed by use of the more precise atomic absorption method for the major cations, Fe, Na, K, and for confirmatory determinations on important minor elements, Mn, Zn, Pb, Ca. Anion contents were determined by classical wet chemical, and ion-selective electrode methods. The principal anions found present were $CO_3^{=}$, $SO_4^{=}$, F^-, and Cl^-. Total N analyses indicated little or no NH_4^+ or NO_3^- ions could be present. Limited X-ray diffraction examinations showed iron oxide as the probable form of iron in the collections.

Comparison of the collections within the Method 5 and in-stack system components show a disproportionate distribution of several anions and cations. Method 5 filter samples contain a higher percentage of iron than the probe, while the probe samples show higher fractions of Na, K, $CO_3^{=}$, and $SO_4^{=}$. The in-stack filter catch contains higher percentages of Fe, Na, and K and a lower percentage of $CO_3^{=}$ than detected in the nozzle particulates. Dissimilarities are also exhibited among the fractions of Fe, Na, K, $CO_3^{=}$, F^-, and Cl^- in the Method 5 and in-stack filter collections. With exception of the in-stack probe data, the sum of the average cation and anion values (Fe and Si calculated as oxides) provides essentially a quantitative account of the composition of the various samples.

Although collections of the two trains (in-stack and Method 5) show fractionation, the differences in composition of major cations and anions in the total system catches when adjusted for weight distribution in the sampling system and normalized to 100 percent (last column of Table 11) are less evident as illustrated in Table 12 below.

TABLE 12. PERCENTAGE DISTRIBUTIONS OF MAJOR CATIONS AND ANIONS WITHIN THE SAMPLING TRAIN COMPONENTS

	Fe	Na	K	$CO_3^{=}$	$SO_4^{=}$	F^-	Cl^-
In-Stack System							
In-stack filter	24.50	4.65	9.60	10.40	3.35	1.45	4.05
Nozzle	2.80	2.52	.42	4.41	.90	.35	.28
Probe	.67	.82	.82	2.70	2.00	.34	.27
Totals:	27.97	7.99	10.84	17.51	6.25	2.14	4.50
Method 5							
Box filter	23.8	2.15	3.8	3.65	2.1	1.44	1.78
Nozzle and probe	4.1	3.80	7.8	9.40	8.0	1.15	3.50
Totals:	27.9	5.95	11.6	13.05	10.1	2.59	5.28

Caution must be taken in drawing firm conclusions from the above tabulations because of the overall averaging and the possible individual analytical errors. Within the cautionary limitations, it appears indicated that, with the exception of $SO_4^{=}$, the overall composition of loadings of the two train systems are similar, i.e., the train configuration and mode of collection does not materially alter the chemical form of the particulate collections. If alterations do occur, it appears indicated that there are somewhat higher contents of Na^+ and $CO_3^{=}$ in the in-stack collections and somewhat higher K^+, $SO_4^{=}$, Cl^-, and F^- in the Method 5 collections.

The general chemical composition of Method 5 samples and a grab sample taken from the BOF/ESP emissions is shown in Table 13. The data, which were obtained by optical emission spectroscopy, do not show any significant compositional differences between Method 5 filter and probe collections and between the Method 5 grab samples.

More quantitative data for several cations and anions in the BOF/ESP samples are presented in Table 14. Cations analyses were performed by atomic absorption spectroscopy, and the anions were determined by classical wet chemistry and ion-selective electrode procedures. The Method 5 filter catches and the grab sample show similarity in composition except for Zn, $SO_4^{=}$, F^-, and C which were found in higher concentrations in the grab sample. Iron predominates the sample contents, accounting for 91+ percent of the weight when assumed to be present as the oxide. The higher carbon value in the probe samples indicates the presence of organics probably introduced in the Method 5 acetone probe washing procedure.

Gaseous Emissions Analysis

The typical composition of the stack gas at the BOF/wet scrubber facility as determined by Orsat and an Environmetrics Faristor monitor are given in Table 15. The gas contains a high concentration of CO which is flared at the top of the stack. Rather high NO_x levels probably result from nitrogen introduced during the blow to purge oxygen from the emission control system. SO_2, if present in the furnace emissions, was probably efficiently removed by the scrubber system and consequently was not detected in the stack gas.

The concentrations of SO_2 and NO_x in the stack gas emission during a typical blow at the BOF/ESP facility are shown in Figure 10. The NO_x level increases steadily during the blow to a maximum level in the range of about 90 to 150 ppm at the termination. The SO_2 level remains fairly constant at a level of about 4 ppm.

Analysis for other gaseous species and organics was performed on samples withdrawn with evacuated glass flasks. The analytical results are reported in Table 16. The primary gas components are CO_2, CO, O_2, and N. SO_2 was detected at approximately the same levels measured with the Environmetrics Faristor. Other sulfur compounds such as H_2S, COS, and CS_2 were not detected (minimum detectable level is 2 ppm). The only organic present at a detectable level was methane.

TABLE 13. ANALYSIS OF PARTICULATE EMISSIONS FROM BOF EQUIPPED WITH ESP CONTROL[a,b]

Element	Filter (3A)	Filter (3B)	Probe (3A)	Probe (3B)	Grab
Fe	40-60	40-60	40-60	40-60	40-60
Si	1	1	1	1	1
Na	0.2	0.2	0.2	0.2	0.2
K	0.5	0.5	0.5	0.5	0.5
Mn	1	1	1	1	1
Ca	1	2	3-4	3-4	3-4
Mg	0.3	0.3	0.3	0.3	0.3
Zn	0.5	0.5	0.5	0.5	0.5
Pb	0.2	0.2	0.2	0.2	0.2
Ni	0.005	0.005	0.01	0.02	0.01
V	0.01	0.0	0.01	0.01	0.01
Cr	0.01	0.01	0.02	0.02	0.01
Co	<0.001	<0.001	<0.001	<0.001	<0.001
Cu	0.03	0.03	0.03	0.03	0.03
B	0.001	0.001	0.001	0.001	0.001
Al	0.02	0.02	0.1	0.3	0.03
Mo	0.01	0.01	0.01	0.01	0.01
Sn	0.003	0.005	0.01	0.02	0.1
Ti	0.005	0.005	0.03	0.03	0.02

(a) Analysis performed by optical emission spectroscopy.

(b) Results in weight percent.

TABLE 14. CHEMICAL ANALYSIS OF PARTICULATE EMISSIONS FROM BOF WITH ESP CONTROL

Sample (Run)	Weight Percent in Sample												
	Fe	Mn	Ni	V	Zn	$SO_4^=$	Cl^-	F^-	$CO_3^=$	C	H	N	P
Method 5 Filter (5A)	70.5	1.27	0.011	0.010	0.43	0.68	0.89	0.28	ND	0.1	<0.1	<0.1	0.03
Method 5 Filter (5B)	71.6	1.28	0.011	0.010	0.44	0.66	0.96	0.64	--	0.1	<0.1	0.1	0.06
Method 5 Probe Residue (5A)	53.8	1.01	0.015	0.008	0.81	0.92	1.15	0.50	ND	7.3	1.0	0.1	0.13
Method 5 Probe Residue (5B)	56.4	1.03	0.017	0.009	1.47	0.84	0.92	0.83	--	5.7	0.8	0.1	0.05
Grab Sample	71.1	1.19	0.010	0.013	0.87	0.98	0.88	1.10	ND	0.4	0.1	<0.1	0.08

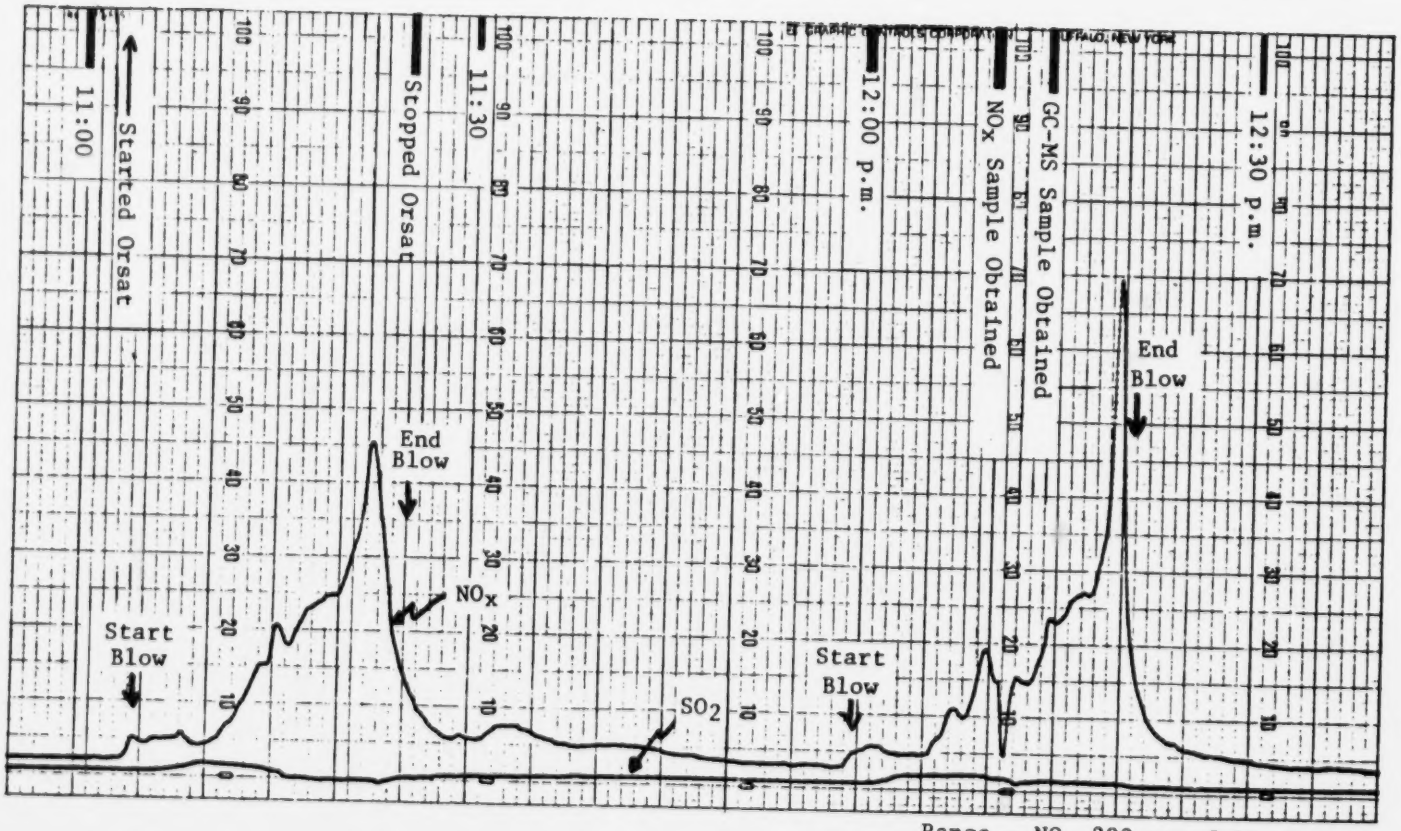
TABLE 15. GAS ANALYSIS OF BOF EMISSIONS WITH WET SCRUBBER CONTROL

Gas	Concentration in Stack Emissions[a]
CO	63 - 81%
CO_2	9 - 20%
O_2	0.1 - 0.3%
NO_x	1034 - 1050 ppm
SO_2	Not detected, >5 ppm

(a) Concentration ranges observed over four heats.

TABLE 16. GAS CHROMATOGRAPHIC AND MASS SPECTROMETRIC ANALYSIS OF GASEOUS EMISSIONS FROM BOF WITH ESP CONTROL

Volume Percent		ppm	
CO_2	12.6 to 13.4	HCl	<2
O_2	12.2 to 13.0	CS_2	<2
CO	0.37 to 0.87	H_2S	<2
N_2	72.3 to 73.0	COS	<2
A	0.93	NO_x	3 to 21
H_2	<0.1		
SO_2	<0.0001 to 0.0004		
CH_4	<0.1		
C_2H_2	<0.1		
C_2H_4	<0.1		
C_3H_8	<0.1		
C_4H_{10}	<0.1		
C_4H_8	<0.1		



Range - NO_x 200 ppm full scale
SO_2 200 pp full scale

Figure 10. NO_x and SO_2 concentrations in BOF emissions during the oxygen blow - BOF with ESP emission control

Impinger Solution Analysis

Impinger solutions from sampling at both BOF facilities were analyzed according to the Method 5 procedure, August 18, 1977. The results shown in Table 17 were obtained.

TABLE 17. RESULTS OF ANALYSIS OF IMPINGER COLLECTIONS

Facility	Run No.	Sample Residue Weight, mgs(a)		
		Water (extracted)	Chloroform/Ether	Acetone
BOF with Scrubber	16A	2.8	1.4	0.3
	16B	4.1	1.4	0.0
	18B	1.8	1.0	0.7
BOF with ESP	3A	4.9	2.0	1.9
	3B	5.1	6.9	0.2

(a) Weights corrected for blanks.

Both the aqueous and organic extract residues gave very low weights. Total impinger catches are approximately 4 to 5 percent of the front end collection for the BOF/wet scrubber and about 4 percent of the front end catch for the BOF/ESP.

Particle Size Measurements

Particle size distribution measurements of the Method 5 and in-stack filter collections from the BOF/wet scrubber emissions are presented in Figures 11 and 12, respectively. The measurements were made by electron microscopic examination of the particulate catches. The data show that the emissions consist of particulates which are primarily submicron in size. Collections by both techniques show essentially the same size distribution with a mean mass diameter of about 0.2 microns.

Particle size distribution measurements of the BOF/ESP facility were made with an Anderson cascade impactor. The mass distribution plotted versus particle diameter is given in Figure 13. Particulate emissions from the ESP are larger in size than from the wet scrubber, the mass mean diameter being about 4 microns.

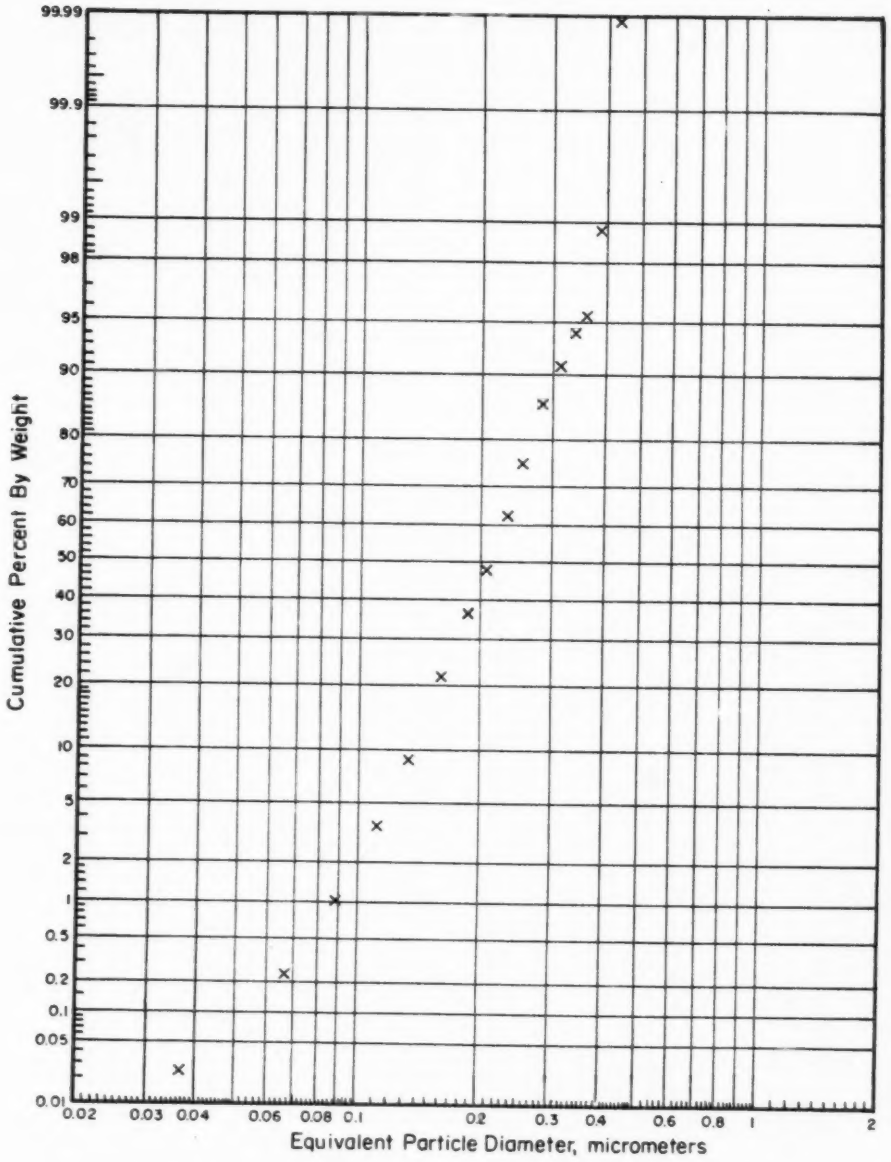


Figure 11. Particle size distribution of BOF/wet scrubber emissions collected on Method 5 filter - run 5B

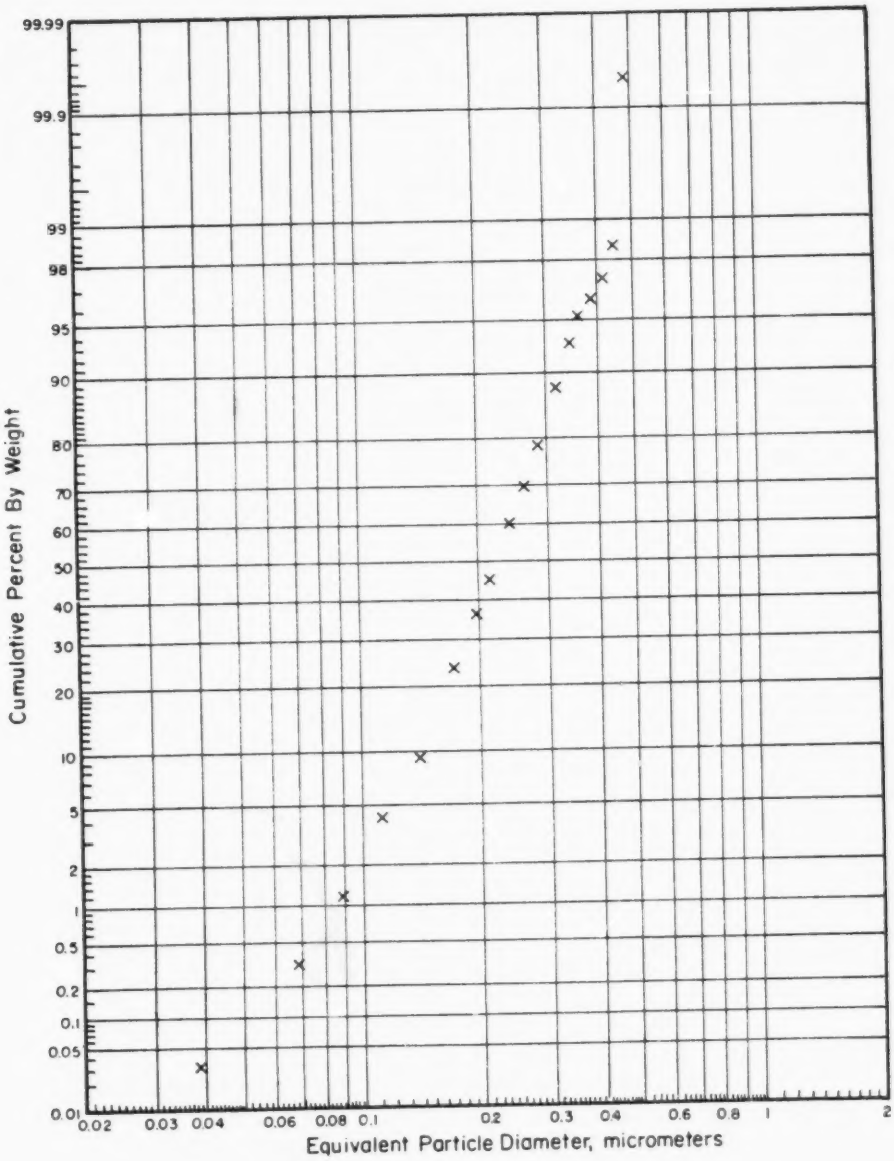


Figure 12. Particle size distribution of BOF/wet scrubber emissions collected on in-stack filter - run 5A

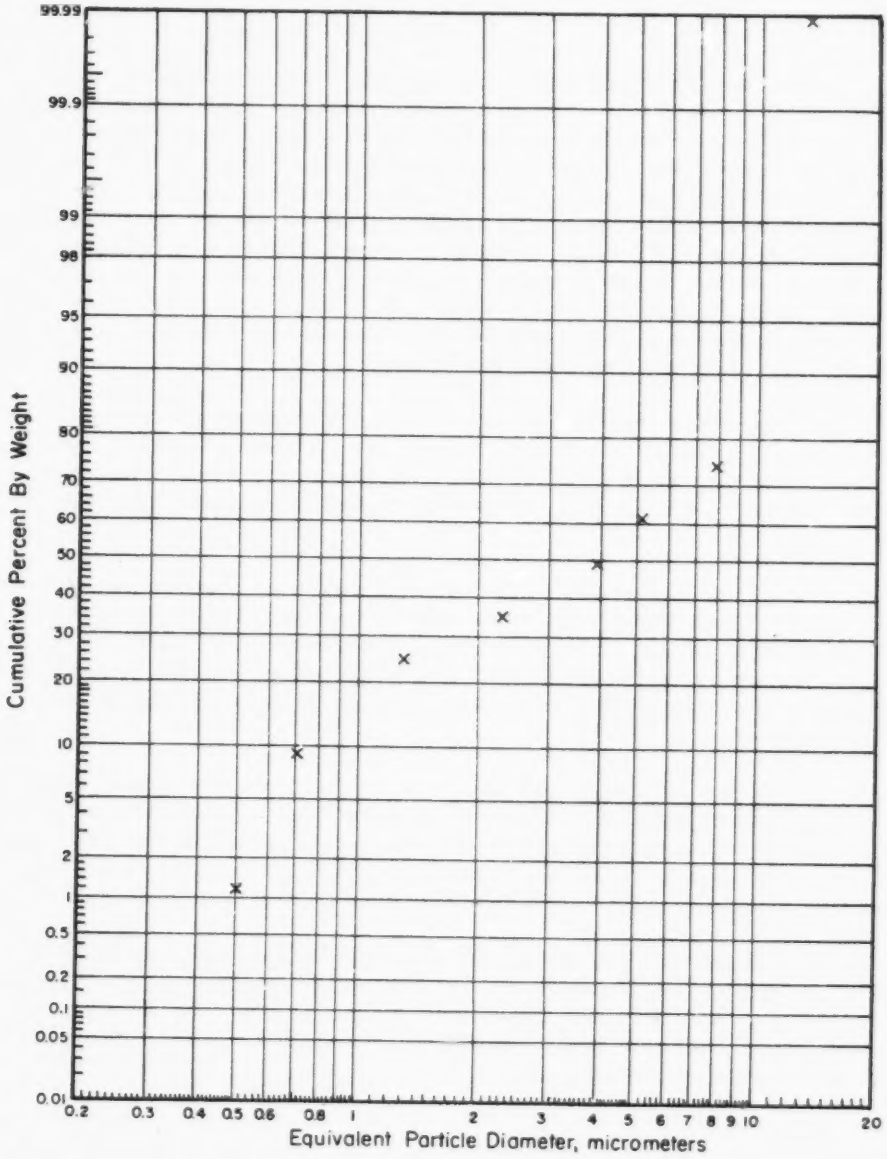


Figure 13. Particle size distribution of emissions from ESP equipped basic oxygen furnace

SECTION 5

DISCUSSION

The study does not indicate any problems associated with the use of Method 5 to determine particulate emissions from basic oxygen furnaces equipped with wet scrubber or electrostatic precipitator controls. Potentially, wet scrubber emissions could present the most difficulty since the sampling system must handle a moisture-laden gas stream with entrained water droplets. If water accumulates on the filter, a high pressure drop will result and limit sample flow. In addition, a wet filter would be more prone to reaction with gas phase species. However, experiments in which the Method 5 sampling train temperature was held at 121°C, the minimum specified temperature, and below, ∿84°C, showed no accumulation of moisture on the box filter.

On the other hand, in-stack sampling presents a formidable problem in maintaining a dry filter during sampling. Without heating, moisture plugs the filter and rupture frequently occurs. External heating may be used with limited success, but adds additional complexity to the sampling process.

The experiments with the BOF/wet scrubber demonstrate that results obtained with Method 5 are not affected by rather large variations in sampling system temperature or deviations from isokinetic sampling rate. Sampling with system temperatures over the range of 84 to 191°C gave results which were in good agreement with sampling performed at the normal Method 5 operating temperature, 121°C.

Variation of the sampling rate at 0.7 and 1.3 times isokinetic also did not significantly affect the accuracy of the mass measurements. The particulate emissions from the scrubber are shown to be very small in size (MMD ∿0.2μ), consequently, deviations from isokinetic sampling would be expected to have a negligible influence on mass measurements.

The chemical characterization work indicates that Method 5 collections are generally representative of the stack particulates. The Method 5 filter collections show the same composition, within the limits of accuracy of the analytical method used, as the particulates removed from the stack by the wet-scrubber system.

In sampling emissions from an ESP equipped BOF, Method 5 was also found to give reliable, reproducible results. Use of a higher purity, lower pH filter medium (ADL quartz) gave mass results which were not statistically different from those obtained with MSA 1106BH, the commonly used filter material. Operation of the sampling system at stack temperature, which would be expected to reduce errors due to condensation reactions, gave mass loading results which were statistically indistinguishable from the values obtained with the train operated at 121°C.

The chemical analyses confirms that the Method 5 procedure extracts a representative sample of the BOF/ESP stack particulates. The same general chemical composition was found in Method 5 filters and grab samples removed from the stack at the sampling point.

Method 5, when compared to two in-stack sampling configurations, appears to give higher mass results. Additional experimentation is necessary to confirm this observation and to identify the source(s) of the discrepancy.

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APPENDIX A

EPA Method 5
Federal Register, December, 1971

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2.1.4 Filter Holder—Pyrex[1] glass with heating system capable of maintaining minimum temperature of 225° F.

2.1.5 Impingers / Condenser—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a ½-inch ID glass tube extending to one-half inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip. A condenser may be used in place of the impingers provided that the moisture content of the stack gas can still be determined.

2.1.6 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° F., dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

2.1.7 Barometer—To measure atmospheric pressure to ±0.1 inches Hg.

2.2 Sample recovery.

2.2.1 Probe brush—At least as long as probe.

2.2.2 Glass wash bottles—Two.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance—To measure to ±0.1 mg.

2.3.4 Trip balance—300 g. capacity, to measure to ±0.05 g.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters—Glass fiber, MSA 1106 BH[1], or equivalent, numbered for identification and preweighed.

3.1.2 Silica gel—Indicating type, 6–16 mesh, dried at 175° C. (350° F.) for 2 hours.

3.1.3 Water.

3.1.4 Crushed ice.

3.2 Sample recovery.

3.2.1 Acetone—Reagent grade.

3.3 Analysis.

3.3.1 Water.

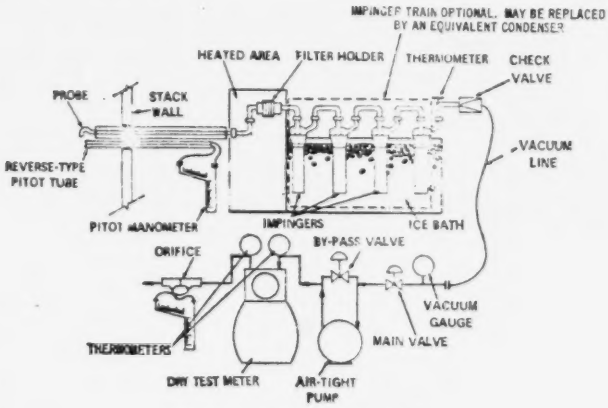

Figure 5-1. Particulate-sampling train.

METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and its weight is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. *Apparatus.*

2.1 Sampling train. The design specifications of the particulate sampling train used by EPA (Figure 5-1) are described in APTD-0581. Commercial models of this train are available.

2.1.1 Nozzle—Stainless steel (316) with sharp, tapered leading edge.

2.1.2 Probe—Pyrex[1] glass with a heating system capable of maintaining a minimum gas temperature of 250° F. at the exit end during sampling to prevent condensation from occurring. When length limitations (greater than about 8 ft.) are encountered at temperatures less than 500° F., Incoloy 825[1], or equivalent, may be used. Probes for sampling gas streams at temperatures in excess of 500° F. must have been approved by the Administrator.

2.1.3 Pitot tube—Type S, or equivalent, attached to probe to monitor stack gas velocity.

[1] Trade name.

[1] Trade name.

[2] Dry using Drierite[1] at 70° F ± 10° F.

3.3.2 Desiccant—Drierite[1] indicating.

4. *Procedure.*

4.1 Sampling.

4.1.1 After selecting the sampling site and the minimum number of sampling points, determine the stack pressure, temperature, moisture, and range of velocity head.

4.1.2 Preparation of collection train. Weigh to the nearest gram approximately 200 g. of silica gel. Label a filter of proper diameter, desiccate[2] for at least 24 hours and weigh to the nearest 0.5 mg. in a room where the relative humidity is less than 50%. Place 100 ml. of water in each of the first two impingers, leave the third impinger empty, and place approximately 200 g. of preweighed silica gel in the fourth impinger. Set up the train without the probe as in Figure 5-1. Leak check the sampling train at the sampling site by plugging up the inlet to the filter holder and pulling a 15 in. Hg vacuum. A leakage rate not in excess of 0.02 c.f.m. at a vacuum of 15 in. Hg is acceptable. Attach the probe and adjust the heater to provide a gas temperature of about 250° F. at the probe outlet. Turn on the filter heating system. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger as low as possible and preferably at 70° F., or less. Temperatures above 70° F. may result in damage to the dry gas meter from either moisture condensation or excessive heat.

4.1.3 Particulate train operation. For each run, record the data required on the example sheet shown in Figure 5-2. Take readings at each sampling point, at least every 5 minutes, and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Sample for at least 5 minutes at each traverse point; sampling time shall be the same for each point. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the rapid adjustments of the sampling rate without other computations. APTD-0576 details the procedure for using these nomographs. Turn off the pump at the conclusion of each run and record the final reading. Remove the probe and nozzle from the stack and handle in accordance with the sample recovery process described in section 4.2.

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
SAMPLE BOX NO. _____
METER BOX NO. _____
METER $\Delta H@$ _____
C FACTOR _____

DIAGRAMMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE _____
BAROMETRIC PRESSURE _____
ASSUMED MOISTURE, % _____
PREDICTED GAS DENSITY _____
PROBE LENGTH, in. _____
NOZZLE DIAMETER, in. _____
PROBE HEATER SETTING _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STACK PRESSURE (P_s), in. Hg	STACK TEMPERATURE (t_s), °F	VELOCITY HEAD (Δp), in.	PRESSURE DIFFERENTIAL across orifice meter (H), in. H_2O	GAS SAMPLE VOLUME (cu. ft.)*	GAS SAMPLE TEMPERATURE AT DRY GAS METER: INLET (Tm in.), °F	GAS SAMPLE TEMPERATURE AT DRY GAS METER: OUTLET (Tm out.), °F	SAMPLE BOX TEMPERATURE, °F	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °F
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 5-2. Particulate field data.

4.2 Sample recovery. Exercise care in moving the collection train from the test site to the sample recovery area to minimize the loss of collected sample or the gain of extraneous particulate matter. Set aside a portion of the acetone used in the sample recovery as a blank for analysis. Measure the volume of water from the first three impingers, then discard. Place the samples in containers as follows:

Container No. 1. Remove the filter from its holder, place in this container, and seal.

Container No. 2. Place loose particulate matter and acetone washings from all sample-exposed surfaces prior to the filter in this container and seal. Use a razor blade, brush, or rubber policeman to lose adhering particles.

Container No. 3. Transfer the silica gel from the fourth impinger to the original container and seal. Use a rubber policeman as an aid in removing silica gel from the impinger.

4.3 Analysis. Record the data required on the example sheet shown in Figure 5-3. Handle each sample container as follows:

Container No. 1. Transfer the filter and any loose particulate matter from the sample container to a tared glass weighing dish, desiccate, and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 2. Transfer the acetone washings to a tared beaker and evaporate to dryness at ambient temperature and pressure. Desiccate and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 3. Weigh the spent silica gel and report to the nearest gram.

5. *Calibration.*

Use methods and equipment which have been approved by the Administrator to calibrate the orifice meter, pitot tube, dry gas meter, and probe heater. Recalibrate after each test series.

6. *Calculations.*

6.1 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2).

6.2 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (70° F., 29.92 inches Hg) by using Equation 5-1.

$$V_{m_{std}} = V_m\left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{P_{std}}\right) = \left(17.71\ \frac{°R}{in.\ Hg}\right)V_m\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{T_m}\right)$$

equation 5-1

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std} = Absolute temperature at standard conditions, 530° R.

T_m = Average dry gas meter temperature, °R.

P_{bar} = Barometric pressure at the orifice meter, inches Hg.

ΔH = Average pressure drop across the orifice meter, inches H_2O.

13.6 = Specific gravity of mercury.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.3 Volume of water vapor.

$$V_{w_{std}} = V_{l_{c}}\left(\frac{\rho_{H_2O}}{M_{H_2O}}\right)\left(\frac{RT_{std}}{P_{std}}\right) = \left(0.0474\ \frac{cu.\ ft.}{ml.}\right)V_{l_{c}}$$

equation 5-2

where:

$V_{w_{std}}$ = Volume of water vapor in the gas sample (standard conditions), cu. ft.

V_{l_c} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

ρ_{H_2O} = Density of water, 1 g./ml.

M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.

R = Ideal gas constant, 21.82 inches Hg—cu. ft./lb.-mole-°R.

T_{std} = Absolute temperature at standard conditions, 530° R.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.4 Moisture content.

$$B_{w_s} = \frac{V_{w_{std}}}{V_{m_{std}} + V_{w_{std}}}$$

equation 5-3

where:

B_{w_s} = Proportion by volume of water vapor in the gas stream, dimensionless.

$V_{w_{std}}$ = Volume of water in the gas sample (standard conditions), cu. ft.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

6.5 Total particulate weight. Determine the total particulate catch from the sum of the weights on the analysis data sheet (Figure 5-3).

6.6 Concentration.

6.6.1 Concentration in gr./s.c.f.

$$c'_s = \left(0.0154\ \frac{gr.}{mg.}\right)\left(\frac{M_n}{V_{m_{std}}}\right)$$

equation 5-4

where:

c'_s = Concentration of particulate matter in stack gas, gr./s.c.f., dry basis.

M_n = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

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PLANT _____

DATE _____

RUN NO. _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			

	VOLUME OF LIQUID WATER COLLECTED	
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
LIQUID COLLECTED		
TOTAL VOLUME COLLECTED		g* ml

CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER. (1 g ml):

$$\frac{\text{INCREASE, g}}{(1\ \text{g/ml})} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

6.6.2 Concentration in lb./cu. ft.

$$c_s = \frac{\left(\frac{1}{453{,}600}\ \frac{\text{lb.}}{\text{mg.}}\right)M_n}{V_{m_{std}}} = 2.205\times10^{-6}\frac{M_n}{V_{m_{std}}} \qquad \text{equation 5-5}$$

where:

c_s = Concentration of particulate matter in stack gas, lb./s.c.f., dry basis.

453,600 = Mg/lb.

M_n = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

6.7 Isokinetic variation.

$$I = \frac{T_s\left[\frac{V_{l_c}(\rho_{H_2O})R}{M_{H_2O}} + \frac{V_m}{T_m}\left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{\theta V_s P_s A_n}\times100$$

$$= \frac{\left(1.667\frac{\text{min.}}{\text{sec.}}\right)\left[\left(0.00267\frac{\text{in. Hg-cu. ft.}}{\text{ml.-°R}}\right)V_{l_c} + \frac{V_m}{T_m}\left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{\theta V_s P_s A_n} \qquad \text{Equation 5-6}$$

where:

I = Percent of isokinetic sampling.

V_{l_c} = Total volume of liquid collected in impingers and silica gel (See Fig. 5-3), ml.

ρ_{H_2O} = Density of water, 1 g/ml.

R = Ideal gas constant, 21.83 inches Hg-cu. ft./lb. mole-°R.

M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_m = Absolute average dry gas meter temperature (see Figure 5-2), °R.

P_{bar} = Barometric pressure at sampling site, inches Hg.

ΔH = Average pressure drop across the orifice (see Fig. 5-2), inches H_2O.

T_s = Absolute average stack gas temperature (see Fig. 5-2), °R.

θ = Total sampling time, min.

V_s = Stack gas velocity, calculated by Method 2, Equation 2-2, ft./sec.

P_s = Absolute stack gas pressure, inches Hg.

A_n = Cross-sectional area of nozzle, sq. ft.

6.8 Acceptable results. The following range sets the limit on acceptable isokinetic sampling results:

If $90\% \leq I \leq 110\%$, the results are acceptable, otherwise, reject the results and repeat the test.

7. *Reference.*

Addendum to Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, Dec. 6, 1967.

Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0581.

Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0576.

Smith, W. S., R. T. Shigehara, and W. F. Todd, A Method of Interpreting Stack Sampling Data, Paper presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14-19, 1970.

Smith, W. S., et al., Stack Gas Sampling Improved and Simplified with New Equipment, APCA paper No. 67-119, 1967.

Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, 1967.

APPENDIX B

STACK GAS MEASUREMENT DATA

TABLE B-1. STACK GAS DATA - BOF WITH WET SCRUBBER EMISSION CONTROL

Run No.	$\sqrt{\Delta P}$ (avg) cmH_2O ½	T_s (avg) C	P_s mm Hg	O_2, %	CO_2, %	CO, %	B_{wo}, %	Md lb/lb-mole	V_s (avg), m/s
1A	0.69	56	745.5	0.2	14	75	20.6	30.2	8.0
B							21.4		
2A	0.97	54	744.7	0.2	14	75	19.4	30.2	11.1
B							16.2		
3A	1.13	54	744.0	0.2	14	75	25.5	30.2	13.2
B							18.9		
4A	0.83	61	749.3	0.2	14	75	30.8	30.2	9.7
B							20.8		
5A	1.00	56	749.3	0.2	14	75	19.0	30.2	11.4
B							20.6		
6A	0.73	64	748.8	0.2	14	75	13.1	30.2	8.2
B							13.4		
7A	0.92	66	749.3	0.2	14	75	17.9	30.2	10.7
B							17.4		
8A	0.86	54	739.4	0.2	14	75	22.0	30.2	9.9
B							17.4		
9A	0.91	53	739.4	0.2	14	75	16.0	30.2	10.2
B							15.0		
10A	0.86	55	747.8	0.2	14	75	16.8	30.2	9.8
B							15.4		
11A	0.94	51	747.8	0.2	14	75	14.3	30.2	10.4
B							13.6		
12A	0.99	58	756.7	0.2	14	75	18.2	30.2	11.2
B							17.2		
13A	0.92	53	751.1	0.2	14	75	20.0	30.2	10.4
B							19.5		

TABLE B-2. STACK GAS DATA - BOF WITH WET SCRUBBER EMISSION CONTROL

Run No.	ΔP (avg), cm H_2O	T_s (avg) C	P_s mm Hg	O_2, %	CO_2, %	CO, %	B_{wo}, %	Md, lb/lb-mole	V_s (avg), m/s
14A	1.00	58	739.6	0.1	14.0	75.0	16.4	30.2	11.54
B							15.9		
15A	0.89	56	745.0	0.1	14.0	75.0	14.9	30.2	10.20
B							15.1		
16A	1.00	59	746.0	0.1	14.0	75.0	19.3	30.2	11.60
B							19.2		
17A	0.89	59	748.8	0.1	14.0	75.0	16.6	30.2	10.32
B							16.0		
18A	1.02	60	748.3	0.1	14.0	75.0	18.2	30.2	11.79
B							17.9		
19A	0.94	84	752.9	0.1	14.0	75.0	15.2	30.2	11.15
B							16.1		

TABLE B-3. STACK GAS DATA - BOF WITH ESP EMISSION CONTROL

Run No.	$\sqrt{P}$ (avg) cm H_2O ½	T_s (avg) C	P_s, mm Hg	O_2 %	CO_2 %	B_{wo}, %	Md, lb/lb mole	V_s (avg) m/s
1A	0.64	133	742.7	17.9	5.5	22.61	29.6	8.2
B						22.78		
2A	0.65	155	734.6	17.9	5.5	25.19	29.6	8.8
B						25.76		
3A	0.64	127	737.1	17.9	5.5	18.91	29.6	8.2
B						18.7		
4A	0.59	144	739.4	17.9	5.5	22.25	29.6	7.7
B						22.51		
5A	0.75	144	734.1	17.9	5.5	16.39	29.6	9.6
B						15.14		
6A	0.56	149	727.5	17.9	5.5	24.93	29.6	7.5
B						25.16		
7A	0.57	153	742.2	17.9	5.5	21.66	29.6	7.6
B						22.08		
8A	0.59	154	745.7	17.9	5.5	21.22	29.6	7.7
B						21.36		
9A	0.59	164	741.2	17.9	5.5	23.06	29.6	7.9
B						22.70		
10A	0.60	147	730.8	17.9	5.5	21.28	29.6	7.9
B						21.24		

APPENDIX C

SAMPLING SYSTEM OPERATION DATA

TABLE C-1. SAMPLING DATA - BOF WITH SCRUBBER

Run No.	Meter Volume (Vm), m^3	Barometer mmHg	ΔH mmH_2O	Avg. Meter Temp (Tm) C	Dry Gas Sampled Std. Cond. (V_{mstd}), m^3	Percent Isokinetic	Average System Temperature, F: Filter box	Gas @ Fltr Outlet	Probe Mid Point	In-stack Filter
1A	1.07	745.5	9.40	29	1.02	86(a)	128	105	126	121
B	1.29		12.85	25	1.25	111	127	128	89	
2A	2.73	744.7	47.50	31	2.60	57(a)	122	123	86	98
B	4.97		118.62	30	4.78	94	126	126	122	--
3A	2.16	744.0	26.92	28	2.08	38(a)	122	119	86	94
B	4.77		136.14	29	4.62	83	136	151	136	--
4A	1.76	749.3	60.20	15	1.78	89(a)	135	116	81	114
B	2.43		86.87	27	2.36	106	134	138	128	--
5A	2.05	749.3	60.96	17	2.06	78(a)	123	133	90	153
B	2.73		123.70	27	2.67	102	135	137	123	--
6A	1.69	748.8	12.09	23	0.74	50(a)	137	131	93	117
B	1.79		17.91	19	1.73	103	127	107	130	--
7A	1.74	749.3	19.30	23	1.71	98	127	123	68	142
B	0.88		14.99	20	1.78	92	129	114	128	--
8A	0.64	739.4	13.21	19	0.63	99	129	119	129	113
B	0.88		14.99	20	0.86	93	128	114	119	--
9A	1.48	739.4	16.76	24	1.42	96	127	118	127	164
B	1.53		15.44	20	1.49	100	131	114	129	--
10A	1.52	747.8	15.37	10	1.56	100	127	118	133	161
B	1.57		14.10	9	1.61	101	128	117	127	--
11A	1.95	747.8	17.53	13	1.97	92	133	116	131	138
B	2.02		16.26	8	2.08	94	123	123	127	--
12A	1.65	756.7	19.91	16	1.68	101	127	118	139	149
B	1.72		18.19	22	1.71	102	125	116	123	--
13A	1.55	751.1	17.27	13	1.58	102	136	118	139	(b)
B	1.60		15.49	14	1.63	104	126	120	124	--

(a) Unable to maintain isokinetic rate due to moisture accumulation on filter.
(b) Thermocouple failed.

TABLE C-2. SAMPLING DATA - BOF WITH WET SCRUBBER EMISSION CONTROL

Run No.	Meter Volume (Vm), m^3	Barometer, mm Hg	ΔH, mm H_2O	Avg. Meter Temp. (T_m), C	Dry Gas Sampled Std. Cond. (V_{mstd}), m^3	Percent Isokinetic	Average System Temperatures, C: Filter Box	Gas at Filter Outlet	Gas at Probe Outlet	Probe Mid-point
14A	2.17	739.6	62.0	22	2.12	104	129	107	130	142
B	2.04		62.5	11	2.08	101	191	180	183	--
15A	1.71	745.0	49.0	11	1.74	105	123	109	126	126
B	1.59		48.3	6	1.65	100	149	148	154	--
16A	1.88	746.0	59.2	17	1.88	105	124	109	128	124
B	1.78		58.9	8	1.83	103	124	111	122	--
17A	1.66	748.8	49.0	13	1.69	100	122	114	131	127
B	2.04		80.5	7	2.12	125	132	131	134	--
18A	1.36	748.3	30.0	18	1.36	74	125	114	127	126
B	1.79		61.5	12	1.83	99	132	133	133	--
19A	0.45	752.9	55.6	14	0.45	105	125	116	131	128
B	0.41			10	0.42	99	171	66	95	--

TABLE C-3. SAMPLING DATA - BOF WITH ESP EMISSION CONTROL

Run No	Meter Volume, (V_m), m^3	Barometer mm Hg	ΔH mm H_2O	Avg. Meter Temp (T_m), C	Dry Gas Sampled Std. Cond. (V_{mstd}), m^3	Percent Isokinetic	Average System Temperatures, C: Filter box	Gas @ Filter outlet	Gas @ probe outlet	Probe Mid-point
1A	1.90	749.8	46.21	27	1.84	105	176	174	179	187
B	1.86			26	1.81	103	122	119	122	128
2A	1.83	741.7	49.02	26	1.76	107	121	114	129	148
	1.83			25	1.77	108	177	168	178	192
3A	1.86	744.2	46.74	17	1.86	100	123	121	123	141
B	1.84			19	1.82	98	177	164	179	178
4A	1.34	746.5	38.86	18	1.34	106	177	149	176	183
B	1.31			17	1.31	104	120	109	128	138
5A	1.42	741.2	60.71	10	1.45	100	126	112	127	144
B	1.38			15	1.39	97	122	103	127	140
6A	1.17	734.6	35.05	19	1.14	109	179	157	179	189
B	1.18			20	1.14	109	177	156	185	179
7A	1.23	749.3	37.34	18	1.23	107	122	110	131	146
B	1.22			17	1.23	107	122	112	129	147
8A	1.19	752.9	39.37	19	1.20	105	121	95	132	147
B	1.17			11	1.21	106	123	114	126	148
9A	1.12	747.8	39.88	16	1.13	110	121	115	131	157
B	1.10		39.88	13	1.12	109	121	108	156	139
10A	1.28	737.9	41.15	21	1.20	105	121	110	126	141
B	1.25			18	1.23	104	121	109	123	143